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During the summer of 1982 the U.S. Environmental Protection Agency (EPA), conducted a comprehensive multimedia environmental monitoring program in the vicinity of two secondary lead smelters located in Dallas, Texas. This monitoring program, which was conducted within a 1 mile radius of the two smelters, included a major soils investigation and the collection and analysis of approximately 3,000 soil, 1,000 blood, and 750 dust samples. Approximately 250 vegetation samples were also collected.

For this study EMSL-LV was requested to provide documentation and guidance for the quality assurance/quality control (QA/QC) program, and to provide the analytical methods and soil sampling procedures protocols. The extensive QA/QC program was applied to all phases of the sampling procedures and analytical methods for documenting the precision and accuracy of the monitoring data. Also, strict chain-of-custody procedures for the sampling and analytical programs were employed to assure the integrity of the monitoring data.

EMSL-LV designed a soil monitoring strategy that identified isopleths of constant soil lead concentration within each of the designated monitoring areas. The results of this soil monitoring strategy plus the protocols, reports and pertinent documentation provided by EMSL-LV for the Dallas Lead Study are presented.

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FOREWORD

This report describes the contribution that the Environmental Monitoring Systems Laboratory at Las Vegas (EMSL-LV), provided to a comprehensive multimedia environmental monitoring study conducted by the U.S. Environmental Protection Agency in Dallas, Texas. The purpose of this study was to:

- Determine the extent of elevated blood lead levels in pre-school age children living near two lead smelters.
- Determine if statistical correlations exist between blood lead levels and certain environmental media lead levels.
- Determine and identify distribution patterns of soil lead concentrations within three designated study areas.

This monitoring program performed under the direction of EPA in Dallas involved the collection and analysis of approximately 5,000 field samples. The precision and accuracy of the environmental measurements obtained were documented through the application of an extensive quality assurance program.

In addition to a brief overview of the study this report presents in chronological fashion the requests received and the assistance provided by EMSL-LV to EPA's regional personnel, the Office of Toxic Substances, the City of Dallas and to the U.S. Public Health Services Center for Disease Control.

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DOCUMENTATION OF EMSL-LV CONTRIBUTION TO DALLAS LEAD STUDY

by

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ABSTRACT

During the summer of 1982 the U.S. Environmental Protection Agency (EPA), conducted a comprehensive multimedia environmental monitoring program in the vicinity of two secondary lead smelters located in Dallas, Texas. This monitoring program, which was conducted within a 1 mile radius of the two smelters, included a major soils investigation and the collection and analysis of approximately 3,000 soil, 1,000 blood, and 750 dust samples. Approximately 250 vegetation samples were also collected.

For this study EMSL-LV was requested to provide documentation and guidance for the quality assurance/quality control (QA/QC) program, and to provide the analytical methods and soil sampling procedures protocols. The extensive QA/QC program was applied to all phases of the sampling procedures and analytical methods for documenting the precision and accuracy of the monitoring data. Also, strict chain-of-custody procedures for the sampling and analytical programs were employed to assure the integrity of the monitoring data.

EMSL-LV designed a soil monitoring strategy that identified isopleths of constant soil lead concentration within each of the designated monitoring areas. The results of this soil monitoring strategy plus the protocols, reports and pertinent documentation provided by EMSL-LV for the Dallas Lead Study are presented.

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INTRODUCTION

During the last part of 1980 a sampling team from the University of Texas at Arlington, under contract with the U.S. Environmental Protection Agency's (EPA) Regional Office in Dallas, Texas, collected samples of soil, paint, house dust, and tap water from a number of private residences and schools located near and in the general vicinity of two secondary lead smelters and one battery plant in Dallas, Texas. In addition, to the two secondary lead smelter sites and the one battery plant sampling site, a fourth site identified as the Reference Area (REF) was also selected for sampling.

All of these sampling sites were located within the Dallas city limits and all except for the REF site were geographically located in the city's western section. As shown in Figure 1, the two lead smelters, RSR Corporation (RSR), and Dixie Metal Company (DMC), are located near the intersections of Westmoreland Road and Singleton Boulevard, and McGowan and Sargent Drive, respectively. General Battery Corporation (GBC) is located near the intersections of Bengal and Amelia Streets. The REF site was selected for its similarity to the RSR, DMC and GBC sites in automotive emissions, population and ethnic background and was located in the city's eastern section near the intersections of St. Augustine and Lake June Roads. The REF site, as shown in Figure 1, was moved at a later date near the intersections of Kiest Boulevard and Frio Drive.

The analytical results of some of the soil samples collected near the RSR and DMC smelter sites showed lead concentrations that exceeded 17,000 ppm. Because of these high soil lead levels, the potential for elevated blood lead levels in pre-school and elementary school aged children living in these areas was considered to be sufficiently high to justify a blood sampling program.

The blood sampling program conducted by the City of Dallas was strictly voluntary in nature and was made available to all citizens living within the Dallas City limits. Nearly 12,000 participants responded to an ambitious program conducted through the media, schools, and door-to-door solicitation.

The EPA volunteered its services to the City of Dallas to assess the quality of the environmental and blood sampling programs previously conducted, evaluate the significance of the data collected and make recommendations for additional monitoring if required. To assist in the evaluation and assessment of the health implications of the blood sampling program, EPA obtained the assistance of the U.S. Department of Health and Human Services Center for Disease Control (CDC), the Texas Department of Health and the City of Dallas Department of Health and Human Services.

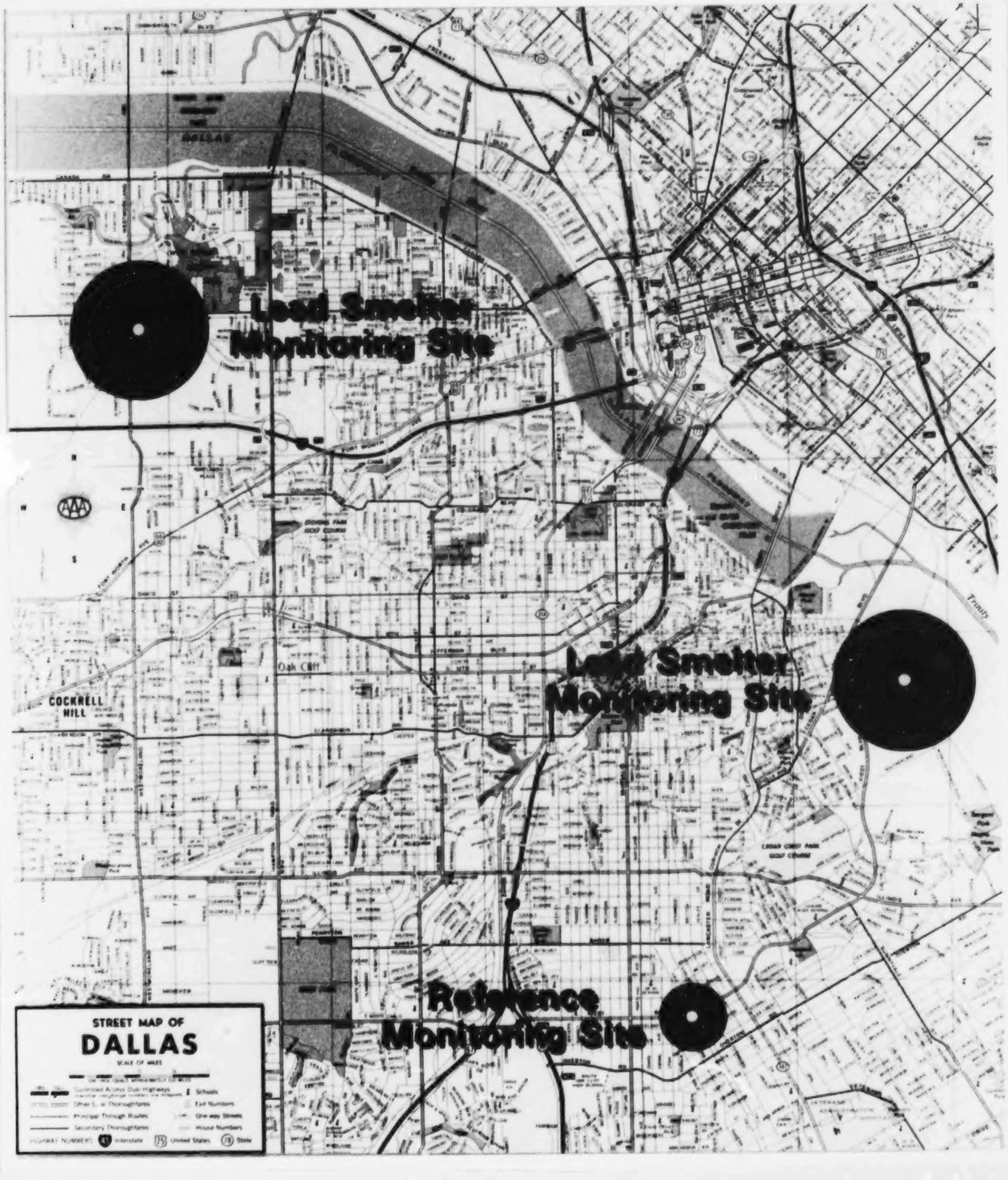

Lead Smelter Monitoring Site
Lead Smelter Monitoring Site
Reference Monitoring Site
STREET MAP OF DALLAS
COCKRELL HILL
Oak Cliff
Trinity

Figure 1. Location of the RSR, DMC and REF sites in Dallas, Texas.

In June 1981, a representative from EPA's Washington Office and a representative from Research Triangle Institute (RTI), an EPA contractor, concluded from a preliminary examination that a lead exposure problem might exist for those individuals living in close proximity to the lead smelters. They also concluded that due to the data deficiencies from the previous studies, it was unclear as to the necessity of initiating any soil cleanup programs. They further recommended that before any costly remedial action was undertaken, the following four facts should be demonstrated:

1. The existence of a potential public health problem in the form of elevated blood lead levels.

2. Elevated blood lead levels around the smelters compared to other areas of Dallas.

3. A decrease in blood lead levels with increasing distance from the smelters.

4. A positive correlation in blood lead levels with soil lead levels.

In September of 1981, the EPA's Env[illegible]mental Monitoring Systems Laboratory in Las Vegas (EMSL-LV) was requested by EPA's Office of Waste Programs Enforcement to assist its Exposure Evaluations Division in reviewing and evaluating the previously collected environmental lead data and to provide input to the EPA's newly-formed Dallas Lead Study group in Washington, D.C.

EMSL-LV was requested to evaluate and comment on the following:

1. Collect, review and evaluate data from the Dallas Lead/Cadmium study.

2. Evaluate sampling techniques, locations and results, and identify deficiencies and strengths. Evaluate the significance of soil sampling depth.

3. Evaluate the analytical work for lead and comment on validity. Identify any weaknesses that might cast doubt on the study results.

4. Using lead data from the Dallas sites and sample locations prepare a map on which is drawn lines of constant lead concentration. Locate the lead plume for the area.

5. Get enough groundwater data (lead analysis) to determine whether or not there is a lead problem in groundwater.

6. Recommend and identify additional monitoring strategies required.

FINDINGS AND RECOMMENDATIONS

In March 1982, representatives from EMSL-LV discussed their findings and recommendations on the environmental sampling methods with a representative of the Office of Waste Programs Enforcement and personnel of EPA's regional staff in Dallas, Texas. The EMSL-LV findings and recommendations are shown in Appendix A. Generally, EMSL-LV scientists found that the sample collection techniques used, the preparation of sample containers, sample packaging, and sample handling were satisfactory. However, there was a total lack of sampling quality assurance for all of the environmental media collected. Sample quality assurance is judged by EMSL-LV to be an essential part of any scientifically designed sampling and monitoring effort. Therefore, EMSL-LV strongly recommended that adequate quality assurance be incorporated into any future monitoring studies.

The tap water lead levels from the DMC, GBC and RSR study sites were not statistically significantly higher than the tap water lead levels collected from the REF study area. Therefore, collection of additional water samples was not recommended.

The dust sampling effort, as designed, could not be used to fully evaluate the indoor respirable particulate fraction. If monitoring in this area is to be continued EMSL-LV recommended the design and initiation of a complementary indoor-outdoor air sampling program.

The soil sampling scheme used by the City of Dallas and the University of Texas-Arlington (Appendix B) was not adequate to delineate geographical areas of lead contamination, nor were sufficient data collected to draw or identify isopleths within any of the study sites. Therefore, a soil monitoring strategy utilizing geostatistics, (kriging), that would provide the necessary data for identifying geographical areas of contamination and isopleths of soil lead levels in each of the study sites was presented to the EPA regional personnel in Dallas (see Appendix A).

Three analytical laboratories, the Civil Engineering Chemical Laboratory at the University of Texas at Arlington which, analyzed soil, dust, and tap water samples; the City of Dallas Environmental Quality Laboratory, which analyzed soil, dust and paint samples; and the ESA Laboratory in Bedford, Massachusetts, which analyzed blood samples, were audited by EMSL-LV personnel during March 1982. The performance audit was restricted to the instrumentation and procedures used for the analysis of lead in samples collected in Dallas and to review the laboratories' QA/QC procedures for evaluating the quality of the analytical lead data generated by each laboratory.

The details of the audit for each of the laboratories are presented in Appendix C. The major problem identified by the EMSL-LV auditors was that the analytical QA/QC procedures used by the Civil Engineering Chemical Laboratory and the City of Dallas' Environmental Quality Laboratory were not adequate to determine the accuracy of the analyses, therefore, the quality of the analytical data could not be determined. Recommendations for correcting these deficiencies are also presented in Appendix C. The procedures and methods used by the ESA Laboratory was judged to be acceptable in all aspects.

Based upon the evaluation of the health implications of the blood sampling program by CDC, the critique and conclusions of the Dallas lead data by RTI, and EMSL-LV evaluation and recommendations concerning the analytical methods and the monitoring strategy, the Dallas Lead Study group concluded that no quantitative relationship could be drawn between soil lead and blood lead levels and recommended that additional monitoring be implemented.

The objectives of this additional monitoring program were to:

1. Determine with precision the blood lead levels in pre-school age children living near two Dallas smelters (RSR and DMC) and in a matched control group.

2. Determine whether a statistical correlation exists between blood lead levels and certain environmental lead levels, and whether inferences can be made regarding those potential sources of lead to blood lead levels in the three study groups.

3. Compare lead levels found in the smelter site study areas with lead levels found in the control area that will permit inferences as to the extent to which blood lead levels in the study areas can be associated with past and present smelter emissions versus other potential sources.

4. Identify the geographical distribution of soil lead concentrations in each study area.

To comply with the Study Group's recommendations and objectives, RTI was asked to design a monitoring strategy (Appendix D, Attachment 4) that would identify the relative contribution of two probable lead sources, lead emissions from automobiles and lead emissions from the RSR and DMC smelters. In addition, RTI's study design was to identify and address the following parameters:

1. Identify the size of the geographical area around each of the smelter sites that should be monitored.

2. Provide traffic pattern data within the recommended monitoring areas.

3. Provide demographic and socioeconomic population data for the study sites.

4. Recommend environmental media that should be sampled to satisfy the objectives.

5. Identify the study population.

6. Determine the number of blood samples required for a statistically valid study in each site.

7. Recommend a suitable REF monitoring site.

In May 1982, EMSL-LV was requested by the Office of Pesticides and Toxic Substances to provide a detailed soil sampling protocol (Appendix D, Attachment 1) that would identify methods of collecting soil samples from which the analytical data could be used to generate isopleths of soil lead concentrations and standard deviations of the estimates of lead concentrations within the RSR, and DMC smelter sites and one designated REF site. The protocol identifies where to collect the samples, the collection methods to be used, the handling and documentation procedures, chain-of-custody requirements for all environmental samples collected, and the soil preparation procedures prior to their shipment to the analytical laboratories. Soil sampling quality control procedures are also identified in the protocol, such as the identification of sampling sites where duplicate samples will be collected. The total number of duplicate, split, field blank and sample bank blank samples required is also identified. In addition, EMSL-LV provided aerial photographs of each of the three sampling sites overlain with the 750' x 750' soil sampling grid network. The soil sampling protocol was extensively reviewed prior to issuance by EMSL-LV scientists, EPA's Region VI, and National Enforcement Investigations Center (NEIC), Denver, Colorado personnel.

EMSL-LV also provided detailed analytical protocols for determining lead in soil and lead in dust samples (Appendix D, Attachment 3). These protocols include procedures for preparing, documenting and analyzing samples, the required quality assurance/quality control (QA/QC) procedures and the format for reporting the results. In addition EMSL-LV provided standard reference materials (SRM) to each analytical laboratory as required.

The analytical protocols were developed after a thorough review of the available literature on lead analysis. The aim was to present methodologies which were consistent with the previous studies, relatively easy to perform, and capable of allowing the analysis of a large number of samples each day. These protocols were also extensively reviewed prior to issuance by EMSL-LV, National Bureau of Standards (NBS), EMSL-Cincinnati, CDC Atlanta scientists, EPA's NEIC, Region VI, and Region I personnel.

The overall QA plan for all field and laboratory operations pertaining to the Dallas Lead Study, except for some components of the blood sampling program was provided by EMSL-LV. The QA plan (Appendix D) identifies the major program elements and the QA objectives of this environmental study. In addition, the following QC procedures, checks, and/or methods are described and documented; Document Control, Chain-of-Custody, Sampling and Analytical Internal QC, Accuracy, Corrective Action, Data Management and System and Performance Audits. All data generated, except for the paint and blood results, were reviewed and validated by EMSL-LV as per the QA plan and further identified and described in Appendix E, QA/QC Procedures for the Dallas Lead Study and in Appendix F, Statistical Analysis of Quality Control Data. Additional information concerning the analysis of soil samples is presented in GCA Volumes I and II titled Analysis of Dallas soil samples GCA-TR-82-104-G December 1982.

During the months of June and July 1982, EMSL-LV was again requested by EPA's Office of Pesticides and Toxic Substances (OPTS), to assist CDC as required and to evaluate their criteria for selecting soil sampling locations in the immediate vicinity of homes, parks and child play areas and to review their (CDC's) dust (Appendix D, Attachment 2), and paint (Appendix G) sampling protocols. EMSL-LV was also requested to submit to OPTS an addendum to the analytical contract task order, contract 68-02-3168, identifying the procedures, methods and requirements necessary to operate and maintain the sample bank. Also required was EMSL-LV evaluation of those analytical laboratory facilities operated by companies interested in providing the soil analysis support as specified in the analytical protocol and in the task order. The facilities and capabilities of two analytical laboratories--GCA of Bedford, Massachusetts, and Envirodyne of St. Louis, Missouri--were evaluated by EMSL-LV. The results of these evalutions are shown in Appendix C.

Prior to the awarding and initiation of the Analytical-Sample Bank Contract, EMSL-LV scientists were required to review and evaluate GCA's work and Quality Assurance Project Plans (Appendix H). The GCA Quality Assurance and Work Plans and their approach for the operation of the sample bank was judged to be statisfactory. GCA was subsequently awarded the contract to provide those two services. In October, GCA, via an amendment to the existing Analytical-Sample Bank Contract, was asked to critique the day-to-day procedures and methods used in conducting the Dallas Lead Study. Their report, with appropriate suggestions for improvement in conducting field studies of this magnitude and scope, is shown in Appendix I.

In June, a five-category land use classification report for the RSR and DMC smelter sites was submitted to EPA's Office of Pesticides and Toxic Substances. This report, (Appendix J), developed by EMSL-LV and Lockheed Engineering and Management Services Company (LEMSCO) scientists for use in implementing any required remedial actions, included the assessment and analysis of information pertaining to the following:

1. Vegetation (includes trees, shrubs, pastures, and naturally occurring grasslands).
2. Water (lakes, ponds, rivers, and other standing water areas).
3. Bare Ground (includes areas mostly devoid of vegetation cover resulting from man's activities and natural processes).
4. Paved Surfaces (includes highways, roads, parking lots and aprons, and storage areas).
5. Buildings/Structures (includes residences, industrial buildings and structures). Acreage (includes lots and land associated with residential development). Residential sidewalks and streets are excluded from this acreage.

In support of both the soil sampling design, and in the operating methods and procedures as identified in the protocols and QA plan pertaining to sampling, sample preparation and handling, chain-of-custody, and in the use of aerial photographs for sample site identification, EMSL-LV personnel assisted EPA's regional staff by providing a list of required equipment and supplies with associated costs and sources of supply and by training personnel. The training program conducted by EMSL-LV included both classroom and field exercises in equipment decontamination procedures, sample identification requirements, collection of QC samples, and in site description documentation requirements and procedures.

Soil and QC field blank samples collected during the field training programs were used to check methods specified in the soil sampling protocol dealing with drying, sieving, and mixing to achieve homogeneity, and to evaluate the effectiveness of the soil auger decontamination procedures. Analytical methods were also evaluated by subjecting a number of samples to both re-extraction and total recoverable procedures. Data obtained from these tests were evaluated before actual field sampling was initiated. The results of these methods tests, as shown in Appendix K, showed the methods to be satisfactory.

The sampling phase of the Dallas Lead Study was initiated on June 23, 1982. From this initial phase through the conclusion of the sampling and analytical programs, EMSL-LV scientists assisted EPA's regional staff in auditing all phases of the field and sample bank operations and exclusively performed all of the analytical laboratory QA/QC performance audits. Appendix L shows the results of the sample bank audit.

During the second week of July 1982, EMSL-LV scientists with assistance from the City of Dallas Health Department opened and operated the sample bank. The sample bank was operated by EMSL-LV personnel for about a two-week period. After this initial period, GCA personnel assumed control of the sample bank operation as per the Analytical-Sample Bank Contract. Audits of the sample bank operation were conducted by EMSL-LV during the study.

The EMSL-LV, in conjunction with LEMSCO performed the function of the referee laboratory as identified in the QA plan. This important QA function involved both data reporting and the analysis of 44 dust and 220 soil samples.

RESULTS

EMSL-LV is required to provide to the Dallas Lead Study group a geostatistical analysis of the lead contamination within each of the study sites. Using the soil lead data obtained from the samples collected from the sampling grid, point kriging of sample parameters will be performed. Point kriging takes into account the continuity of sample data as expressed by the calculated variograms, the location of the samples around the point, and their clustering. The level of lead present is estimated at each point and a precision value is given for each estimate.

As further described in Appendix M, the kriging results will be displayed as contour maps showing estimated lead levels within all three study sites. These maps will be accompanied by kriged error maps indicating the confidence level of the estimation.

Appendix N shows the results of the geostatistical analysis. The use of geostatistics by the EPA's EMSL-LV for the Dallas Lead Study has promoted understanding, use, and application of geostatistics for evaluating environmental contamination.

APPENDIX A

CRITIQUE OF ENVIRONMENTAL LEAD MONITORING STUDIES CONDUCTED IN THE VICINITY OF SECONDARY LEAD SMELTERS IN DALLAS, TEXAS

DALLAS LEAD STUDY

INTRODUCTION

Data collected by the City of Dallas and the University of Texas at Arlington have shown some soils collected in the immediate vicinity of the Dixie Metal Company (DMC) and the RSR Corporation (RSR) have lead levels that exceed 1,000 µg/g. A sampling scheme was designed and implemented by both entities that identified lead levels in a variety of media including tap water, house dust and house paint in the areas surrounding these two industrial facilities. Due to the behavior of airborne lead from industrial facilities such as these, the sampling area was confined to a 2-mile radius around each of the sites. To evaluate the degree of contamination of these two industrially exposed sites, a site identified as the reference area (REF) was selected near the intersections of Lake June and St. Augustine Roads. This reference area was judged by available windrose data to be out of the probable deposition pattern of the industrial sites. The sampling scheme, except for the total number of samples collected in the reference area, was identical to that used at the DMC and RSR sites. In addition, a 2-mile area surrounding a third industrial site, the General Battery Corporation (GBC), was sampled using the same scheme as previously mentioned.

Sampling Scheme

Sampling sites were selected using a stratified random sampling design. The study perimeter included all of the area lying within a 2-mile radius of the suspected contaminant source. From the center of the study area, 16 equally-spaced radii (subtending an angle of 22.5°) were drawn. The study area was further divided by 20 concentric circles spaced from the center at 0.1 mile increments. Each of the resulting sampling sectors were coded by direction and distance from the center. The sectors were selected for sampling by standard random techniques with the following restrictions:

1. At least two-thirds of all sampling sectors were to be in the major downwind direction (Wind Rose Data From Dallas Love Field 1961-1973).
2. Residential and non-residential sites must exist in the sector sections.
3. Sampling sectors were judged eligible for repeat selection only if they were geographically located within 1/2-half mile from the center of the sampling area or if the sector contained both residential and nonresidential sites.
4. Twenty (20) soil sampling locations were selected.
5. Ten (10) of the locations were selected at residences and ten (10) were located at nonresidential sites, such as, schools, parks, playgrounds and/or daycare centers.
    a. Cold and hot tap water was collected from the residential sites.

    b. Dust was collected from residential sites which included swipes and samples from dust collecting trays.
    c. Paint chip samples were collected.

The selection of the specific site within the sampling sector was determined by the on-site investigator. Guidelines used included:

1. Permission of home owners
2. Physical biases
    a. Sites judged too close to major roads were not selected.
    b. Obvious contaminated areas (oil spills, etc.) were not selected.

SAMPLING METHODS

Soils

A maximum of six and a minimum of four sampling sites were selected at each residential location. Two of the samples were collected in the front yard, two in the backyard and two in the garden, if present, at each residence. At the nonresidential sites the number of sampling locations were selected in part by the activity patterns of children since they were judged to be the critical receptors.

A stainless steel corer with an inside diameter of 3/4-inch was used to collect the soil. The corer, marked in 1-inch increments to a depth of 3 inches was pushed into the soil. After the corer was withdrawn, the soil was removed by a plunger and disected into three 1-inch depth increments. Each increment was treated as a separate sample.

Documentation of sampling locations included site description forms showing the specific sampling site relative to structures, trees and roads. Photographs were also taken of some of the sampling sites.

Guidelines used in sampling site selections included:

1. Samples collected were spaced at least 20 feet from any painted surface.
2. Sampling sites were located as far from areas of vehicle activity as possible, i.e., streets, driveways and parking areas.
3. Sampling sites selected were at least 4 feet from structures in which paint chips might occur.
4. Collection of samples under or immediately adjacent to trees and shrubs were avoided when possible.
5. Sampling sites were selected to represent conditions of the yard, i.e., if yard was barren of growth the site was selected in a barren area.

The techniques used by the City of Dallas and the University of Texas, Arlington, to collect the soil samples are commonly used by soil scientists, as such, the use of the soil corer was judged to be satisfactory. The extraction of soil from the corer, the disection of soils into the 1-inch increments and the subsequent packaging and handling of the soil samples was also judged to be satisfactory.

Poor sampling techniques were judged to be in the following areas:

1. Soil Variation - No attempt was made to obtain a representative sample or to evaluate the wide variation in lead levels that existed in a number of soils collected from the same site. For example, two soil samples collected from a residential lot at 225 Slade Street contained lead levels of 564 and 4,909 μg/g, respectively. Soil lead levels reported from a number of other sites were also of the same magnitude of variability.

2. Cross contamination - The sampling equipment was washed and dried after use at each site. The washing and decontamination of sampling equipment is mandatory for the prevention of sample cross contamination. However, there was no attempt to evaluate or identify whether the washing and/or drying technique selected was adequate. Swipes and samples of rinsings should have been collected and analyzed.

TAP WATER

Both hot and cold tap water was collected in the kitchens of each residence. The tap was opened for a minimum of 1 minute before any of the samples were collected. The samples were collected in 500 ml acid-washed polyethylene bottles. Prior to sample collection the bottles were rinsed three times with either hot or cold tap water. After collection the samples were acidified (1%) within 90 minutes using concentrated reagent grade HNO_3. The techniques used to collect tap water samples, their packaging and handling, are accepted methods for the collection of water samples for metal analysis.

As far as can be determined from the available data, sampling of domestic water occurred only one time. Also, it appears that duplicates or split samples were not collected.

There was no significant difference in the lead levels in cold tap water between any of the study areas (GBC, DMC and RSR) and the REF study area. Also, the data shows that there was no significant difference in lead levels in hot tap water samples collected from the GBC, REF, and DMC study sites. However, the hot tap water lead levels were significantly lower in RSR study site samples than those from the GBC, REF and DMC study sites. As such, collection of additional water samples is not recommended.

DUST AND PAINT

Swipes of household dust were collected from windowsills and tops of refrigerators. Where applicable, swipes were collected from windowsills of inside play areas and children's bedrooms. To supplement swipes, a dust collecting tray was placed upon the refrigerator top for a 28-day period.

Swipes of household dusts were collected by two methods. One involved the use of a commercial towelette (Dioparene) and the other was a Kimwipe moistened with distilled water. After collection, the moistened collectors were placed in sample containers until analyzed. The dust collection tray was an acid-washed rectangular polyethylene container. After the 28-day collection period the tray was sealed with an acid-washed polyethylene lid until analyzed.

Paint chip samples were collected from residential areas which showed flaking on the walls or windowsills. A small pocket knife was used to collect approximately 1 gram of paint chips. The paint chips were placed in plastic containers until analyzed.

As per citizen's request, outdoor dust swipes were collected. The collection of outdoor dusts were primarily confined to automobiles. However, one 8-foot canopy was also swiped.

The location of the swipe samples was recorded. Also, the dimensions, approximately 100 cm^2 of the area swiped, was measured and recorded.

Swipes and dust collecting devices are commonly used and may provide needed information concerning exposures of particulate contaminants by the inhalation pathway. However, in an active household in which significant volumes of indoor and outdoor air are mixed, the heavier particulates would tend to settle out first, thereby possibly biasing the data obtained from this sampling technique. Using only this method, adequate assessment cannot be made for the smaller and more respirable particulate fraction. For example, studies reported by Boggess 1977 have shown that particles with diameters of less than 10 µm are not significantly affected by gravitational settling. Also, particulates less than 10 µm are generally considered to be the most respirable.

Analysis of the dust collecting media, Dioparene towelettes and Kimwipes, should be completed to identify and evaluate their lead content and their suitability as dust sample collectors.

If warranted, a monitoring scheme should be designed that will address outdoor and indoor air sampling complemented by dust sampling. In addition to total lead analyses of these samples, analysis should include size determination and chemical characterization.

SOIL SAMPLING DESIGN

The Environmental Monitoring Systems Laboratory in Las Vegas was requested to review the collected soil data, draw lines of constant concentration of lead (isopleths), critique the monitoring scheme used, and recommend additional soil monitoring if necessary.

Data collected from previous soil sampling studies have indicated that higher lead concentrations were generally found in those soils collected in close proximity to the suspected source. As distance increased from the suspected source the soil lead levels generally decreased. Also, the data indicates that a depth migration of lead by some means, probably mechanical, has occurred. However, the sampling scheme used is not adequate to delineate geographical areas of lead contamination, nor was there sufficient data collected to adequately draw or identify isopleths. Therefore, even though requested, no isopleths were drawn.

The data was analyzed by a one-way analysis of variance. These analyses, as shown in Appendix A, show that the reference area (REF) mean lead level is statistically significantly lower at the 95% confidence level than the GBC mean lead level. Also, the GBC mean lead level is significantly lower at the 95% confidence level than the mean lead levels of both the DMC and RSR sites. In addition, the data shows that there is no statistically significant difference at the 95% confidence level existing between the mean lead levels of the DMC and RSR sites. However, 1,000 μg/g of lead is above the 99% confidence interval of the mean of either the DMC or RSR sites.

In summary, these data show that the DMC and the RSR sites belong to a different and higher population of lead levels than the GBC or the REF sites. All sample means were less than 1,000 μg/g and statistically significantly below 1,000 μg/g. Thus, since GBC is significantly lower than either DMC or RSR additional sampling may be justified in the RSR and DMC sites but no additional sampling is justified in the GBC site.

Maps of the four study areas were made. Soil lead levels reported by the City of Dallas and the University of Texas at Arlington were plotted showing the approximate collection locations and the mean values calculated from the surface soils collected at each location. The mean levels were then ranked as a function of distance from the suspected source. The information obtained from this ranking was used to construct a semi-variogram for each of the study areas as shown in Appendix B. Using this data and the statistical methodology of kriging, sampling grid dimensions were calculated. Statistically, kriging is designed to make use of the dependence of one sample with other closely associated samples and is able to produce unbiased estimates from the data. It not only will estimate the values of spatially distributed variables but will also identify the probable error associated with the estimates.

The calculated and recommended sampling scheme to identify the geographical extent of the soil lead contamination is as follows:

1. The dimensions of the square grid should be 750' x 750'. The total area covered by the grid will be contingent upon the minimum lead concentrations of concern. The grid should also be oriented if possible along the axis of the plume.

Based on the plotted data for the DMC study area, it appears that soil levels similar to REF levels would be found if the grid extended north of Ervay and Beaumont Streets, and south of Southerland and Bonnie View Road intersections. The western boundary should extend to Highway 342 and east of Highway 45. Examination and identification of lead from nearby major vehicle arteries may require an eastward extension of the grid to Highway 75. The outer limits of the grid can be extended or decreased as warranted. Extension of the grid to the west may be necessary due to the high lead levels 731 and 1,249 µg/g found at DMV-11. These two values should be verified by reanalysis of extract if available or by additional samples before extending the grid.

The soil lead levels appear to be more localized in the RSR study area. As such, soil levels similar to REF levels would probably be encountered if the grid extended 1.0 miles to the east, west, and southern directions and 1.5 miles in the northern direction from the RSR facility. Extension of the grid can be increased or decreased as warranted.

It is recommended, as previously suggested, that sampling be terminated at the GBC site as the soil data doesn't indicate excessive lead levels in this area.

It is recommended that the REF grid dimensions (750' x 750') be identical to that used for the DMC and RSR sites. The geographical location of the REF site appears to be satisfactory.

2. Each of the grid intersections should be identified by map coordinates for future data interpretation. Soils within a radius of 30 meters of each of the grid intersections should be sampled using procedures identical to those described by the City of Dallas. The 30-meter radius from the grid intersections will provide on-site flexibility for selection of suitable sampling sites. The use of the core sampler and the sample handling and chain-of-custody technique used should be adhered to. Data received by EMSL-LV does not show or indicate any problems were encountered in these areas with soil-organic layers. As such, only mineral soil should be collected. Also, only the upper 3 inches should be collected (see Appendix C). If organic or vegetative layers are commonly

encountered a sampling scheme would have to be designed to address this parameter. The on-site investigator shall be responsible for determining the specific sampling site. Criteria used for making this determination will be identical to those previously used and identified in the University of Texas sampling protocol. Documentation at each site shall include photographs showing sampling area, a schematic showing sampling locations, including dimensions and other pertinent information including structures, canopy layers, ground surface characteristics, streets, driveways, etc.

Following the selection of a specific sampling area, four subsamples should be collected. As noted in Appendix D, calculation of the in-yard variance of the surface soil samples was less than the between-yard variance suggesting that four subsamples per sampling site is adequate. The subsamples should be collected 15 meters from the center of the sampling area, one each from the north, south, east and west directions. The corresponding depth increments should be placed in appropriate sample containers and mixed prior to analysis. To obtain a homogeneous sample, it is recommended that mixing be completed in the laboratory.

We recommend that sample integrity be maintained as per EPA NEIC chain-of-custody requirements, and that recommended sample containers, preservation and holding time requirements be adhered to as per the Federal Register, Vol. 44, No. 233, 1979.

PRECISION AND ACCURACY

It is recommended that precision and accuracy of the sampling procedures be addressed. The degree and level of precision and accuracy is dependent on the ultimate use of the data. Recommended additional samplings for addressing precision and accuracy include:

1. Collection of duplicate samples.
2. Collection and analysis of splits.
3. Analysis of spikes and field blanks.
4. Decontamination samples, such as equipment swipes, rinse water and/or solvents used in equipment cleaning.

Literature Cited

1. Boggess, R. W. Editor. Lead in the Environment. 1977. Chapter 7. Transport and Distribution from Mining, Milling and Smelting Operations in a Forest Ecosystem, p. 139. Prepared for the National Science Foundation Research Applications Directorate.

APPENDIX A

Pollution observations are often from a logarithmatic normal rather than a normal distribution. To test which distribution is Dallas' lead, the skewness was calculated. The skewness was improved by the log transformation and the transformed data was used in the analysis of variance and other statistical tests.

The original sampling had two aberrant observations in DMC at 14-HS. These were resampled and the new observations replaced the original values.

The analysis of variance showed statistical significance (at 95%, 98%, 99% levels) between areas.

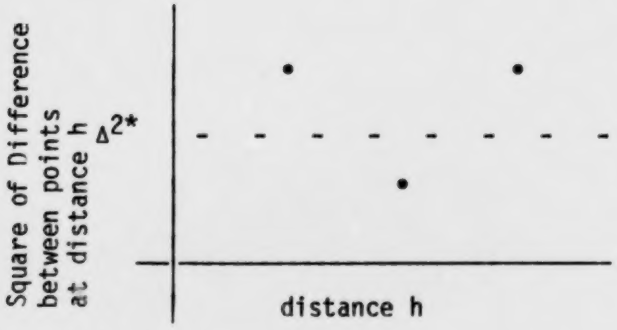
Duncan's multiple range test shows three populations as diagrammed below:

REF GBC DMC RSR

At the 95 percent level DMC and RSR are from one population of lead observations shown by one underscore, but GBC and REF are different populations discrete from each other and each represented with its own underscore.

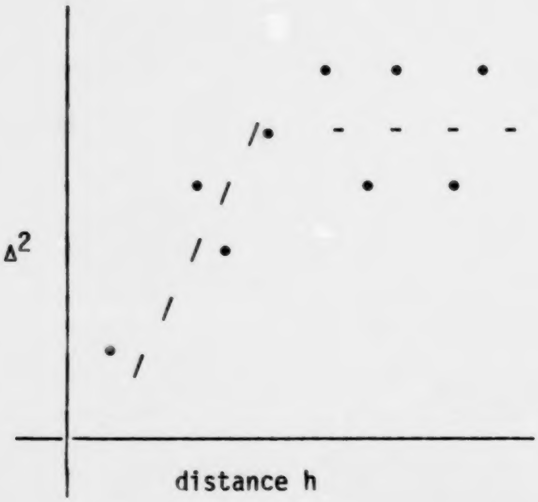
APPENDIX B

How far apart should the samples be? Classical statistics deals with random variables and computes how many samples should be taken. Geostatistics deals with regional (or spatial) variables and computes how far apart samples should be taken. A regional variable is autocorrelated in time or space or both. Thus, a lead observation is a regional variable if a sample value is numerically closer to spatially nearby samples and is numerically different from remote samples. Geostatistics examines this correlation structure by means of a semi-variogram. This is a graph of the square of the difference between samples at distance h apart plotted against distance h. If the samples are uncorrelated the variogram is a horizontal line. Interestingly, the variogram of REF was a horizontal line. This indicates that the lead



*(square of difference in concentration between pairs of all points at distance h)

values found in REF were not structured. This is what should be found in a control area. If the samples are correlated, the first values of h-distance have rising or increasing squares of differences. At greater h-distances their squares of difference become horizontal as in the previous variogram. The first or rising part of the plot is the informative part because it measures the range of correlation. This is the distance before the plot becomes horizontal. The distance between sampling points must be about 2/3 of the range of correlation. Interestingly, both DCM and RSR had this type of semi-variogram (called spherical or Metron model). This shows there is structure or autocorrelation to the lead samples in these sites. The ranges of correlation at the two sites were about 1,200 feet and thus the recommended grid distance between sampling points is 750 feet.



APPENDIX C

How deep a plug should be taken for a sample? It is most important that all samples must be taken with pluggers of identical inside diameter and depth. Obviously all samples will be compared for national background levels or world averages. This requires uniformity. However, since nine profiles with lead content reported inch by inch of depth were done in Dallas, this opportunity was taken to evaluate the use of 3-inch plugs.

Studying the data, the first three depths (1"-3") are representative of higher values. In general, lower depths (4"-6") drop off in lead content. Since lead is usually in insoluble forms this is expected. DMC-14-HS-04 and DMC-17-HS-01 are exceptions to this generalization because their highest values are at inch four; however, even in these exceptions, inch five belongs to the lower range of lead content. Cultivation or other disturbances would physically explain these anomalies. A one-way analysis of variance shows the lead values of the top 3 inches to be statistically significantly higher at 99 percent confidence level than the lead values of the bottom 3 inches.

APPENDIX D

How many samples should be composited to describe a sampling site? This is a very important question and should be answered in terms of spatial variables (see Appendix B). Because of the time limit, this question was answered in terms of random variables. Within an area (e.g. DMC, RSR) the between-site-variance was statistically significantly larger at the 95 percent level than the within-site-variance. A two-way analysis of variance tested this. This implies that additional effort should be invested initially in sampling more sites in order to reduce the between site variance since it is the larger. This implies that the within-site-variance is adequately described by four (4) samples per site.

APPENDIX B

THE CITY OF DALLAS AND THE UNIVERSITY OF TEXAS AT ARLINGTON SAMPLING AND ANALYTICAL PROTOCOLS

Sample Selection and Analysis Protocol

"Lead and Cadmium Monitoring In The Dallas, Texas Area"

(OT-918-NTEX)

Sample sites were selected using the following procedure. First a survey was conducted by driving and walking through the area to determine the location of elementary schools, day care centers, playgrounds and home residences. The survey was conducted within a two mile radius of each plant site.

The study area was then divided into sectors that radiated outward from the plant under study. The sectors were twenty two and one half degrees wide and aligned such that the wind directions (indicated by the wind rose for Love Field, Dallas) bisected the sectors. The sectors were further delineated into approximately one tenth mile sections using concentric circles whose radii varied by one tenth of a mile. Each segment was coded by direction and outward distance. A random number generator produced a directional number and then an outward distance number. Numbers generated were used under the following restrictions:

1. Two thirds of all sites must be in the major down wind directions.
2. Residences and non-residence sites must exist in the sector section.
3. Sectors were not allowed to be repeated unless they were within one half mile of plant or that sector section contained both residence and none residence sites.

Selection of an individual site within a section sector was accomplished by the person doing the sampling. Residences were selected on the basis of some one being home and willing to participate in the sampling program. Non-residence sites were selected on the basis of their existance within a sector section. Large areas within each plant area often contained neither residence nor non-residence sites. Sites that were obviously biased by their selection were rejected by the sampler. These biases include sites that were adjacent to major traffic arteries or other contaminent sources.

Soil samples were collected using the following guide lines:

1. Two samples were collected from both the front yard and the back yard where they existed.

2. Samples collected were spaced at least twenty feet from any painted surface.

3. Individual sample sites were located as far from areas of vehicle activity as possible, ie. streets, drive ways and parking areas.

4. Collection under or immediately adjacent to trees and shrubs were avoided when possible.

5. Individual soil samples were selected to represent conditions of yard, ie. if yard was barren of growth, sample was selected from barren spot and vise versa.

6. Obvious low or high spots were avoided as sample sites.

7. Soil samples were extracted directly from samples and placed directly into polyetholene container without being handled by person collecting sample.

PROTOCOL FOR THE SELECTION OF SOIL LEAD SAMPLING SITES

The three inch (3/4" diameter) core sampling technique was selected after preliminary sampling studies indicated a need for soil strafication analysis. This technique allowed the sampling analysis of the top, middle and bottom inch of the soil for lead content. Since persons are basically exposed to the top inch of soil, this method was feasible. Likewise, analysis of the second and third inches allowed us to examine the extent of lead contamination.

Sites were chosen based on the following considerations:

1. Resampled sites from EPA lead study.
2. Citizens request.
3. Schools, playgrounds and day care centers within the two mile radius of the lead smelters (RSR and Dixie).
4. Follow-up and expanded sampling of sites where high soil lead levels were indicated from the first lead study.
5. Expanded area sampling (random) to determine more accurately the distribution of lead contamination within a given radius. This allowed the City to create an orderly sampling pattern which would help establish the extent of soil lead.

SOIL SAMPLING SITE SELECTION:

When applicable, soil samples from residential sites were taken following the criteria listed below:

1. 50 feet away from the street (usually on the side yard and

back yard). This method eliminated influence from automobile lead emissions.

2. Four or more feet from the house where the influence of paint chips might occur.
3. Area of the yard where children might be exposed through playing.
4. Away from any area that was littered or contained signs of parked cars or automotive work.

Soil samples from schools, play areas and day care centers within the two mile radius of the two smelters were selected from:

1. Areas where children played.
2. Areas where children ate outdoors.

These sites were selected because of the possibility of exposure to a large population within a central location. Preference was given to elementary schools and day care centers because of the age groups involved. These age groups (9mo. - 8yrs.) constituted a higher risk catagory due to the playing and pica habits.

DUST WIPES SELECTIONS:

The criteria for selection of residence where wipes were taken is listed below:

1. Citizen request for lead analysis.
2. Follow-up study where previous lead analysis indicated high soil lead.
3. Residential follow-up of persons with high blood lead levels as indicated by the City's screening program.

Where applicable, dust wipes were taken from the window sills of the inside play area and subject's bedroom and from the top of the refrigerator. On two request, dust wipes were taken from two parked cars and an 8 foot canopy.

The bases for taking paint chip samples were also centered around the above forementioned criteria. Paint chip lead analysis became important where young children resided, since the liklihood of ingested paint chips increased.

SAMPLING PROCEDURE

MATERIALS (Soil)

1. Stainless steel laboratory 3/4" diameter cork bore & plunger with a 3" depth mark indicator.
2. Sterile sealed whirl-pack plastic containers
3. Small spaterla (for reaming soil stuck in sampler)
4. Drying towel
5. Water for washing equipment
6. Labels
7. Materials for drawing & mapping the sample sites
8. Gloves
9. 6" x 9" envelopes

MATERIAL (Dust Wipes)

1. Laboratory kimwipes (small)
2. Sterile Whirl-pack plastic containers
3. Laboratory bottle containing distilled H_2O
4. Gloves
5. 6" x 9" envelopes
6. Labels

SOIL SAMPLING

1. After the site was selected using the above mentioned criteria, the cork bore was pushed into the soil to a 3" depth and withdrawn.

2. The plunger was inserted and the soil evacuated 1" at a time and placed in the labeled whirl-pack plastic containers. The containers were sealed according to the instructions included in the whirl-pack package, and placed in a labeled envelope (6" x 9") and sealed. The envelope contained a total of 6 soil samples taken from 2 sites in the residential yard.
   The plastic containers were labeled with name, address, site number, and ABC indicated A-top inch, B-middle inch and C-bottom inch of extracted soil.
3. All used equipment (cork bore) was washed, dried and placed back in the sample case before use at the next site.
4. All soil samples were kept in a locked truck until custody was transferred to the lab at the end of a sampling day. A map was drawn detailing the area where the soil samples were taken, and the type of soil extracted.

DUST WIPE PROCEDURES

1. The kinwipe was moistened using the distilled H_2O contained in the laboratory bottle.
2. The selected areas wipes (window sills, refrigerator,etc.) contained approximately 100 cm^2. The used wipe containing the sample was placed in the whirl-pack plastic container, labeled and sealed. All wipe samples were placed in a labeled envelope and kept in a locked truck until custody was transferred to the lab at the end of a working day.

3. The results from the lab were given in ug which were then converted into ug/cm^2.
4. If the window sill used was not 100 cm^2, the results were adjusted using the correct window sill measurements taken.

PAINT CHIP SAMPLING PROCEDURES

1. Paint chip samples were taken from residential areas which showed flaking, on the walls or window sills.
2. A small pocket knife was used to obtain the chips from the selected areas.
3. The chips (approximately 1 gr.) were placed in a labeled whirl-pack plastic container and sealed. These samples were also placed in a labeled envelope and transferred to the lab for analysis.
4. The knife used for gathering the sample was washed before being used again.

Memorandum

CITY OF DALLAS

DATE: October 19, 1981

TO: James T. Blankenship, Director

SUBJECT: Lead Extraction from Soil Samples

Attached is a copy from the Federal Register, Volume 43, No. 194, which describes lead extraction and analysis for air samples collected on a high volume filter. Our laboratory is using the hot extraction method described in Section 7.2 for soil samples after performing the following sample preparations:

1. Dry the soil sample overnight in a 105° F oven.
2. Grind dried samples to powder with glass mortar and pestal.
3. Sieve powdered sample through a coarse screen.
4. Weigh a one gram aliquot of the sample in a 150 ml beaker.
5. Continue procedure described in 7.2.12 thru 7.2.1.5.9 of Federal Register.

Al Harris, Supervisor
Environmental Quality Laboratory

RECEIVED
NOV 27 1981
Pesticides Branch
Dallas, Texas

of HNO_3 and hydrochloric acid (HCl) facilitated by ultrasonication.

1.3 The lead content of the sample is analyzed by atomic absorption spectrometry using an air-acetylene flame, the 283.3 or 217.0 nm lead absorption line, and the optimum instrumental conditions recommended by the manufacturer.

1.4 The ultrasonication extraction with HNO_3/HCl will extract metals other than lead from ambient particulate matter.

2. *Range, sensitivity, and lower detectable limit.* The values given below are typical of the methods capabilities. Absolute values will vary for individual situations depending on the type of instrument used, the lead line, and operating conditions.

2.1 Range. The typical range of the method is 0.07 to 7.5 μg Pb/m³ assuming an upper linear range of analysis of 15 μg/ml and an air volume of 2,400 m³.

2.2 *Sensitivity.* Typical sensitivities for a 1 percent change in absorption (0.0044 absorbance units) are 0.2 and 0.5 μg Pb/ml for the 217.0 and 283.3 nm lines, respectively.

2.3 Lower detectable limit (LDL). A typical LDL is 0.07 μg Pb/m³. The above value was calculated by doubling the between-laboratory standard deviation obtained for the lowest measurable lead concentration in a collaborative test of the method.*(15)* An air volume of 2,400 m³ was assumed.

3. *Interferences.* Two types of interferences are possible: chemical and light scattering.

3.1 *Chemical.* Reports on the absence *(1, 2, 3, 4, 5)* of chemical interferences far outweigh those reporting their presence, *(6)* therefore, no correction for chemical interferences is given here. If the analyst suspects that the sample matrix is causing a chemical interference, the interference can be verified and corrected for by carrying out the analysis with and without the method of standard additions.*(7)*

3.2 Light scattering. Nonatomic absorption or light scattering, produced by high concentrations of dissolved solids in the sample, can produce a significant interference, especially at low lead concentrations. *(2)* The interference is greater at the 217.0 nm line than at the 283.3 nm line. No interference was observed using the 283.3 nm line with a similar method.*(1)*

Light scattering interferences can, however, be corrected for instrumentally. Since the dissolved solids can vary depending on the origin of the sample, the correction may be necessary, especially when using the 217.0 nm line. Dual beam instruments with a continuum source give the most accurate correction. A less accurate correction can be obtained by using a nonabsorbing lead line that is near the lead analytical line. Information on use of these correction techniques can be obtained from instrument manufacturers' manuals.

If instrumental correction is not feasible, the interference can be eliminated by use of the ammonium pyrrolidinecarbodithioate-methylisobutyl ketone, chelation-solvent extraction technique of sample preparation.*(8)*

4. *Precision and bias.*

4.1 The high-volume sampling procedure used to collect ambient air particulate matter has a between-laboratory relative standard deviation of 3.7 percent over the range 80 to 125 μg/m³.*(9)* The combined extraction-analysis procedure has an average within-laboratory relative standard deviation of 5 to 6 percent over the range 1.5 to 15 μg Pb/ml, and an average between laboratory relative standard deviation of 7 to 9 percent over the same range. These values include use of either extraction procedure.

4.2 Single laboratory experiments and collaborative testing indicate that there is no significant difference in lead recovery between the hot and ultrasonic extraction procedures.*(15)*

5. *Apparatus.*

5.1 Sampling.

5.1.1 High-volume sampler. Use and calibrate the sampler as described in reference 10.

5.2 Analysis.

5.2.1 Atomic absorption spectrophotometer. Equipped with lead hollow cathode or electrodeless discharge lamp.

5.2.1.1 Acetylene. The grade recommended by the instrument manufacturer should be used. Change cylinder when pressure drops below 50-100 psig.

5.2.1.2 Air. Filtered to remove particulate, oil, and water.

5.2.2 Glassware. Class A borosilicate glassware should be used throughout the analysis.

5.2.2.1 Beakers. 30 and 150 ml. graduated, Pyrex.

5.2.2.2 Volumetric flasks. 100-ml.

5.2.2.3 Pipettes. To deliver 50, 30, 15, 8, 4, 2, 1 ml.

5.2.2.4 Cleaning. All glassware should be scrupulously cleaned. The following procedure is suggested. Wash with laboratory detergent, rinse, soak for 4 hours in 20 percent (w/w) HNO_3, rinse 3 times with distilled-deionized water, and dry in a dust free manner.

5.2.3 Hot plate.

5.2.4. Ultrasonication water bath, unheated. Commercially available laboratory ultrasonic cleaning baths of 450 watts or higher "cleaning power," i.e., actual ultrasonic power output to the bath have been found satisfactory.

5.2.5 Template. To aid in sectioning the glass-fiber filter. See figure 1 for dimensions.

5.2.6 Pizza cutter. Thin wheel. Thickness <1mm.

5.2.7 Watch glass.

5.2.8 Polyethylene bottles. For storage of samples. Linear polyethylene gives better storage stability than other polyethylenes and is preferred.

5.2.9 Parafilm "M".* American Can Co., Marathon Products, Nennah, Wis., or equivalent.

6. *Reagents.*

6.1 Sampling.

6.1.1 Glass fiber filters. The specifications given below are intended to aid the user in obtaining high quality filters with reproducible properties. These specifications have been met by EPA contractors.

6.1.1.1 Lead content. The absolute lead content of filters is not critical, but low values are, of course, desirable. EPA typically obtains filters with a lead content of <75 μg/filter.

It is important that the variation in lead content from filter to filter, within a given batch, be small.

6.1.1.2 Testing.

6.1.1.2.1 For -large batches of filters (>500 filters) select at random 20 to 30 filters from a given batch. For small batches (<500 filters) a lesser number of filters may be taken. Cut one ¾"x8" strip from each filter anywhere in the filter. Analyze all strips, separately, according to the directions in sections 7 and 8.

6.1.1.2.2 Calculate the total lead in each filter as

$$F_b = \mu g\ Pb/ml \times \frac{100\ ml}{strip} \times \frac{12\ strips}{filter}$$

where:

F_b = Amount of lead per 72 square inches of filter, μg.

6.1.1.2.3 Calculate the mean, $\bar{F}_b$, of the values and the relative standard deviation (standard deviation/mean × 100). If the relative standard deviation is high enough so that, in the analysts opinion, subtraction of $\bar{F}_b$, (section 10.3) may result in a significant error in the μg Pb/m³ the batch should be rejected.

6.1.1.2.4 For acceptable batches, use the value of $\bar{F}_b$ to correct all lead analyses (section 10.3) of particulate matter collected using that batch of filters. If the analyses are below the LDL (section 2.3) no correction is necessary.

6.2 Analysis.

6.2.1 Concentrated (15.6 *M*) HNO_3. ACS reagent grade HNO_3 and commercially available redistilled HNO_3 has found to have sufficiently low lead concentrations.

6.2.2 Concentrated (11.7 *M*) HCl. ACS reagent grade.

6.2.3 Distilled-deionized water. (D.I. water).

6.2.4 3 *M* HNO_3. This solution is used in the hot extraction procedure. To prepare, add 192 ml of concentrated HNO_3 to D.I. water in a 1 *l* volumetric flask. Shake well, cool, and dilute to volume with D.I. water. *Caution:* Nitric acid fumes are toxic. Prepare in a well ventilated fume hood.

6.2.5 0.45 *M* HNO_3 This solution is used as the matrix for calibration standards when using the hot extraction procedure. To prepare, add 29 ml of concentrated HNO_3 to D.I. water in a 1 *l* volumetric flask. Shake well, cool, and dilute to volume with D.I. water.

6.2.6 2.6 *M* HNO_3+0 to 0.9 *M* HCl. This solution is used in the ultrasonic extraction procedure. The concentration of HCl can be varied from 0 to 0.9 *M*. Directions are given for preparation of a 2.6 *M* HNO_3+0.9 *M* HCl solution. Place 167 ml of concentrated HNO_3 into a 1 *l* volumetric flask and add 77 ml of concentrated HCl. Stir 4 to 6 hours, dilute to nearly 1 *l* with D.I. water, cool to room temperature, and dilute to 1 *l*.

6.2.7 0.40 *M* HNO_3 + X *M* HCl. This solution is used as the matrix for calibration standards when using the ultrasonic extraction procedure. To prepare, add 26 ml of concentrated HNO_3, plus the ml of HCl required, to a 1 *l* volumetric flask. Dilute to nearly 1 *l* with D.I. water, cool to room temperature, and dilute to 1 *l*. The amount of HCl required can be determined from the following equation:

$$y = \frac{77\ ml \times 0.15\ x}{0.9\ M}$$

---

*Mention of commercial products does not imply endorsement by the U.S. Environmental Protection Agency.

University of Texas at Arlington

Chemical analysis quality control was accomplished using the following:

1. A continuous source dual beam atomic absorption sepestrophotometer (AAS) with automatic background correction was usual at a 283.3 nm. wave length to minumize interference from light scattering.

2. All glass ware (borosilerate) and polyethalene used was washed in laboratory detergent, rinsed, soaked for twenty four hours in concentrated nitric, rinsed in distilled/dionized water and dried.

3. All samples (both liquid and solid) were stored in linear polyethaline containers.

4. AAS was dynamically calibrated using working solutions that were prepared from National Bureau of Standards certified standard reference solutions. AAS was calibrated only on linear ranges. Samples outside linear ranges were diluted and rerun until within linear ranges.

   AAS was checked for reproducability of standard every tenth sample. A ten percent variation (plus or minus five percent) in working solution standard reading required the recalibration of AAS and rerun of last ten samples.

5. Both analytical and Reagent blanks were run. They were run on the basis of five percent of the total samples run. These blanks revealed no detectable lead or dadmium from reagents or hardware used in the analysis. (Baker ultra pure reagents for trace metal analysis were used.)

6. Replication of ten percent of all samples was performed to assure that concentration found were reliable.

7. Spiked samples were used to determine recovery effeciency on all samples. Analysis of spiked samples were performed equally by both persons during analysis work. Spiked samples were done on a basis of five percent of the total number of sampler. Recovery factors determined were applied to all AAS findings. Recovery factors standard deviations were less than ten percent the mean recovery factors.

APPENDIX C

ANALYTICAL LABORATORY EVALUATIONS

U.S. ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS
P.O. BOX 15027, LAS VEGAS, NEVADA 89114 • 702/798-2100 (FTS 545-2100)

Date:

Reply to Attn of: QAD

Subject: Evaluation of the Department of Civil Engineering Chemical Laboratory, University of Texas, Arlington, Texas, March 25, 1982

To: Files

PERSON CONTACTED:	Professor Vittorio K. Argento, University of Texas, Arlington
EVALUATION TEAM:	Dr. Werner F. Beckert, EMSL-LV Dr. Steve Simon, LEMSCO, Las Vegas
EVALUATION OBSERVER:	Mr. Edwood Martin, OWPE, EPA HQ

PURPOSE: To evaluate the Department of Civil Engineering Chemical Laboratory, University of Texas, Arlington, Texas (henceforth called UTA Laboratory) with respect to analyses performed for the Dallas Lead Study, and to review the available QA/QC information in order to define the quality of the analytical lead data generated by the UTA Laboratory for the Dallas Lead Study.

BACKGROUND: The UTA Laboratory team, consisting of Dr. Ernest C. Crosby and Mr. Vittorio K. Argento, both professors at the Department of Civil Engineering, University of Texas, Arlington, TX, collected soil, dust and tap water samples from the vicinity of two active lead smelters in Dallas and from a reference area, extracted the samples and analyzed the extracts for lead and cadmium using atomic absorption spectrophotometry (AAS). This task was conducted under contract for Region 6, contract number OT-0918-NTEX. In the contract, the sampling and QA approach were outlined in general terms, whereas the extraction and analysis methods were specified in detail. According to the contract, EPA approval of the protocol and the QA plan was required. The contract did not include requirements for outside analysis of a representative number of samples nor for total lead determination in any sample. Maintaining chain of custody was not required. Copies of pertinent pages of the contract are enclosed as Appendix 1.

Dr. Crosby and Mr. Argento jointly collected the samples. Dr. Crosby then extracted the samples at the UTA Laboratory using a mixture of acetic acid and hydroxylamine hydrochloride, and Mr. Argento and Dr. Crosby jointly analyzed the extracts for lead at the Texas Air Control Laboratory, Austin, TX, using flameless AAS. A total of 232 soil samples from 60 sites near smelters, and 54 soil samples from 54 sites in a reference area, were collected and analyzed. Dr. Crosby and Mr. Argento conducted the total task without additional help.

This evaluation is restricted to items pertaining to the analysis of Dallas soil samples for lead by the UTA Laboratory, as required by the above contract. Unfortunately Dr. Crosby was hospitalized at the time of the laboratory evaluation. Also, the AAS unit is located in Austin, Texas and was not inspected. Accordingly, a number of details pertaining to sample preparation and analysis could not be verified.

A copy of a Laboratory Evaluation Checklist used during the evaluation is enclosed as Appendix 2.

The evaluation team wishes to acknowledge the excellent cooperation provided by Professor Argento during this laboratory evaluation.

PERSONNEL: The team conducting this task of the Dallas Lead Study consisted of Dr. Crosby and Mr. Argento as equal team members. Both Dr. Crosby and Mr. Argento are qualified researchers, as evidenced by their résumés (Appendix 3). Mr. Argento has been involved in earlier lead studies when he was working for the City of Dallas. The Crosby/Argento team has conducted two follow-up lead/cadmium studies, both under contract to EPA, Region 6. Copies of the report title pages are enclosed as Appendix 4.

FACILITIES & EQUIPMENT: During a tour of the UTA Laboratory it was verified that the laboratory facilities were adequate. There was sufficient laboratory and bench space available and a fume hood in good condition. Deionized water of adequate purity was available, as well as laboratory equipment required for the task (drying oven, desiccators, hot plates, shaker, glassware, class A volumetric flasks, etc.). Although it was not required under the contract that this task was to be carried out following chain-of-custody guidelines, the samples and extracts, when not in use, were always kept in a secured storage area. General housekeeping was good.

PROTOCOLS & DOCUMENTATION: No separate written sample receipt, storage, sample extraction and sample analysis operating procedures exist, however, due to the small number of people involved in this task (two) and due to their experience and qualifications it is probable that the integrity of the samples was preserved at all times. The numbering system used by the team is adequate. Mr. Argento had no problems in answering any of our questions about sample receipt, storage and extract analysis.

No general QA plan exists for the UTA Laboratory. No QA officer had been formally nominated, and the team did not routinely participate in interlaboratory studies (e.g., EMSL-CIN water analyses). However, the team has been involved in other studies that required analysis of soil samples for lead (Appendix 4).

No special QA plan had been prepared for this study, however, details of the proposed QA were discussed with the Region 6 Project Officer, and his consent was apparently obtained. The following quote on quality assurance as performed for this task has been taken from the final report, dated February 24, 1981, page 14:

> "Quality assurance procedures included: the use of reagent blanks; determination of recovery factor for each type of extraction; replication of ten percent of all AAS readings; maintenance of AAS linear analysis region by using calibration curve and monitoring standard solutions every tenth sample at a minimum; and the diluting of samples if outside linear concentration region."

A copy of the final report is enclosed as Appendix 5.

The data generated for the task were recorded on loose sheets which were then assembled in folders. An inspection of the records showed that they were adquate. Each team member was responsible for maintaining his own records.

During all AAS analyses carried out by the team, both team members were present. Mr. Argento performed the analyses while Dr. Crosby recorded the results. Ten percent of all extracts were analyzed in duplicate, Dr. Crosby performing the analyses of the duplicates and Mr. Argento recording the results. Both team members reviewed all data generated.

To the best of Mr. Argento's recollection, a logbook exists for the AAS at the Austin facility for recording performance information such as calibration data and service activities.

CONTAMINATION CONTROL: Glassware (borosilicate) and polyethylene equipment used for this task were washed in a laboratory detergent, rinsed with distilled/deionized water, soaked for 24 hours in concentrated nitric acid, rinsed with distilled/deionized water and air dried.

All samples (both soils and extracts) were stored in linear polyethylene containers.

High-purity reagents (Baker Ultrapure™) were used for the trace metal analyses.

Analytical and reagent blanks were analyzed on a five-percent basis of the total number of samples prepared and analyzed. These blanks revealed no detectable lead contamination from the reagents or equipment used in the analysis, as verified by a review of the records.

ATOMIC ABSORPTION CALIBRATION AND METHODOLOGY: An IL Model 251 Atomic Absorption Spectrophotometer with automatic background correction was used in the flame mode for the lead-in-soil determinations. The wavelength used was 283.3 nm.

The AAS was calibrated using working standards that were prepared from "NBS certified" reference solution (Fisher Scientific Company). The working lead standards were prepared in HNO_3.

The AAS was calibrated and used only in the linear range (up to 15 ppm Pb). Whenever the lead value obtained for a sample was $\geq$ 15 ppm, the sample was diluted to a concentration below 15 ppm and reanalyzed.

AAS calibration was rechecked by analyzing a calibration standard after every tenth sample. A ±5 percent drift required recalibration and reanalysis of the last ten samples.

All samples were extracted once; ten percent of the extracts were reanalyzed by a different operator to check AAS result reliability. No duplicate samples were extracted and the extracts analyzed.

Five percent of the soil samples were spiked with a standard solution of $Pb(NO_3)_2$, extracted and analyzed for lead. Notebook data revealed that the recovery factors determined for the lead extraction averaged approximately 75 percent and had a standard deviation from the mean of less than ten percent. The recovery factors were used to adjust all acquired lead data to a 100-percent recovery basis before reporting.

DISCUSSION: Personnel qualifications, facilities and equipment of the UTA Laboratory seem to be adequate. The lack of formal detailed operating procedures for sample receipt, storage, sample extraction and sample analysis can be explained by budgetary restrictions and by the small size of the laboratory team. Detailed discussions with Mr. Argento about various aspects of this task indicated to the evaluation team that the absence of formal operating procedures probably did not influence the quality of the data generated.

A detailed written QA plan prepared for this study would definitely have been very valuable, if only because a careful review would have shown areas of deficiencies. The simple statement pertaining to quality assurance in the final report is not adequate.

The procedures for contamination control used during the sample preparation and analysis phase were adequate for this task. The laboratory facilities were found to be in adequate condition to minimize sample contamination. This was also evidenced by the fact that no lead was detected in both analytical and reagent blanks.

The instrumental procedures used for calibration were documented in the project notebook and found to be consistent with manufacturer's guidelines. The calibration standards were prepared from NBS-certified reference solution and can therefore be assumed to be reliable, and an instrumental drift check was performed at an adequate frequency (10 percent). A corrective action procedure existed for excessive drift (±5 percent).

The laboratory (Austin) where the atomic absorption work was performed was a certified laboratory for air pollution analyses and had a record of adequate performance on interlabortory PE samples (this was stated by Mr. Argento but was not verified by the evaluation team).

Unfortunately, no laboratory quality control samples such as NBS-traceable reference standards or EPA QA samples were analyzed during this task to actually verify calibration accuracy. Assuming that the certified stock lead solutions had not been contaminated and the working solutions were properly diluted (this was verified from the investigator's notebook), then it is safe to assume that the calibration standards were accurate, or at least of a quality sufficient for a survey study where trends are of importance.

Working standards were prepared in HNO_3, and an independent analysis (using the same working standards) was performed on ten percent of the extracts. A data check revealed that results agreed within ten percent of the original analysis.

These results indicate that the precision of the AA analysis was within ±10 percent which is acceptable for this task.

The extraction precision is not known. It could have been determined by extracting a representative number (10 to 15 percent) of duplicates.

The accuracy achieved for the analysis of the soil extracts can not be determined from the data available. The method of analysis used, i.e., direct comparison to a working curve without use of the method of standard addition, does not allow a direct evaluation of the effect of the sample matrix (extractant plus soil extract) on lead absorption in the flame. The matrix effect could be positive or negative, depending on the nature of the matrix. The impact of the matrix effect on observed AAS results is commonly evaluated using the method of standard addition.

The only control which seems to pertain to matrix effects in this task is the extraction of soil samples that had been spiked with $Pb(NO_3)_2$ solution, followed by AAS analysis of the extracts. It was found that consistently (±10 percent RSD) 75 percent of the amount of lead added to the soil samples was recovered, in terms of analysis by AAS, from the extract. The analysis results of real samples were therefore corrected to a 100-percent recovery basis before reporting. However, the observed (based on AAS results) incomplete recovery of spikes could be either the result of incomplete extraction, e.g., because of absorption to the soil, or it could be due to an effect of the sample matrix on lead absorption in the flame, or both; therefore, a correction of the analysis results to a 100-percent recovery basis does not seem justified. Since, as explained in the preceding paragraph, the effect of the sample matrix on lead absorption in the flame has not been determined, an "observed" recovery, in terms of analysis by AAS, of 75 percent (with good precision) could in reality be higher (e.g., 90 percent) when the matrix effect is negative, or lower (e.g., 50 percent) when the matrix effect is positive. Therefore it is imperative that the matrix effect be determined before any valid extraction efficiency statements can be made.

It is not known what fraction of the total soluble lead was removed by a single extraction. This could have been determined by repeated extractions of a number of soil samples, perferably from different areas. There is therefore no assurance that the reported values represent the total extractable lead. Spiking of soil samples with $Pb(NO_3)_2$ followed immediately by extraction and analysis does not necessarily give an answer since $Pb(NO_3)_2$ is soluble and should be readily extracted with the extractant used (acetic acid/hydroxylamine hydrochloride) unless some undefined adsorption or reaction occurred with the sample which would render part of the spiking lead insoluble. Furthermore, nothing is known about the chemical form of lead in the samples, and therefore about the solubility, and it is not known whether or not the lead species present in the soil varied from location to location.

The contract did not specify analysis of samples for total lead, and consequently no total lead determinations were performed. However, it could be worthwhile to analyze at least 10 to 15 samples for total lead (e.g., by complete digestion using $HClO_4/HF$, or by x-ray fluorescence) to determine the percentage of extractable lead. It would further be worthwhile to have a similar number of samples extracted according to the method (HNO_3 extraction) used by the Environmental Quality Laboratory, City of Dallas, and the extracts analyzed using the method of standard addition. This effort could provide comparability between the extraction methods used by the UTA Laboratory and the Environmental Quality Laboratory, City of Dallas.

The composition of soil samples from different sampling locations may differ. This would impact on extractability of lead and on matrix effects. Dr. J. B. Corley, Assistant Professor, UTA, a geologist who is very experienced in soil characterization has evaluated some of the soil samples collected by the team for this task. She classified all of the samples she had evaluated thus far as silty clay/clayey silt, with some organic material. This information seems to indicate that any variation of sample composition would probably be minor and would not result in drastically different extractability of the samples.

CONCLUSIONS: Based on the above it is not possible to determine the degree of accuracy of the lead-in-soil analyses, and therefore the quality of the analytical data, from the information available. The data can be used for the detection of relative differences between the sampling sites for extractable lead, however, no absolute value for extractable lead (with a defined accuracy) can be assigned to any one sample. Additional studies (detailed below) would provide more information on the accuracy of the data. However, the UTA Laboratory data can not be correlated with the data generated by the Environmental Quality Laboratory, City of Dallas since the soil samples used by the UTA Labortory team were aliquots from 3-inch long cores whereas all but 50 of the samples analyzed by the Environmental Quality Laboratory consisted of only the top one-inch segment of the soil core.

RECOMMENDATIONS: Since Dr. Crosby and Mr. Argento have sufficient soil material available, the following actions are recommended:

1. Fifteen soil samples collected under this task should be sent to Dr. Tom Spittler, Region 1, who has agreed to analyze them for total lead content via x-ray fluorescence techniques. Dr. Spittler has demonstrated to the evaluators that he has extensive experience in determining total lead in soil samples using this and other methods. The samples selected should include three samples from the reference area. The remaining 12 samples should fulfill the following requirements, if possible:

    a. come from different areas;

    b. should encompass the range of soil variation encountered during the study (one criterion for selection might be the color of the soil sample);

    c. should include samples that resulted in high and low lead values for their respective extracts.

2. If available, the extracts from the above soil samples should be reanalyzed for lead, using the method of standard addition, to determine the matrix effect.

3. To provide comparability between the two different extraction methods, splits of the 15 samples should be extracted using the extraction method as applied by the Environmental Quality Laboratory. Preferably these extractions should be performed at the Environmental Quality Laboratory. The extracts should then be analyzed via AAS using the method of standard addition.

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Date:

Reply to Attn of: QAD

Subject: Evaluation of the Environmental Quality Laboratory, Environmental Health and Conservation, City of Dallas, Dallas, Texas, March 17 and 18, 1982

To: Files

PERSONS CONTACTED:	Mr. Gary Rogan, Laboratory Manager Mr. Fred Barnes, City of Dallas
EVALUATION TEAM:	Dr. Werner F. Beckert, EMSL-LV Dr. Steve Simon, LEMSCO, Las Vegas
EVALUATION OBSERVERS:	Dr. Daniel Paschal, CDC, Atlanta Mr. Edwood Martin, OWPE, EPA HQ Mr. Ray Lozano, QA Officer, Region 6

PURPOSE: To evaluate the Environmental Quality Laboratory, Environmental Health and Conservation, City of Dallas with respect to soil analyses performed for the Dallas Lead Study and to review the available QA/QC information in order to define the quality of the analytical lead data generated for this task.

BACKGROUND: As part of the Dallas Lead Study, personnel from the Department of Environmental Health and Conservation, City of Dallas, collected soil, swipe, paint and food samples in the vicinity of two active lead smelters in Dallas, extracted/digested the samples in the Environmental Quality Laboratory (EQ Laboratory) and analyzed the extracts for lead and cadmium using atomic absorption spectrophotometry (AAS). Blood samples were collected from individuals in those areas and sent to ESA Laboratories, Bedford, Massachusetts, for lead determination.

In a follow-up study, additional soil samples were collected in the areas where individuals with high blood lead levels are living, and analyzed for lead. Six to eight samples, consisting of soil, dust, wipes, and maybe fruit or vegetables, were collected per site and transferred to the EQ Laboratory. The EQ Laboratory has thus far received and analyzed sample sets from 135 locations. An outline of the follow-up study is enclosed as Appendix 1.

The average routine workload for the EQ Laboratory includes analysis of approximately 20 to 25 surface water samples per week (to be analyzed for coliforms, nitrate, turbidity, pH, conductivity, BOD), and approximately 30 food samples per week for bacterial analysis (a microbiologist is in charge of this part). The State Health Department certified the EQ Laboratory for bacteriological analyses and for the operation of hi-vol air samplers; it also periodically certifies individuals for certain activities pertaining to bacteriological aspects. The bacteriological section was integrated with the EQ Laboratory in October 1981.

Since the main mission of the laboratory is to maintain and calibrate an air monitoring network, more data on air pollutants are collected than on anything else. Accordingly, the analysis of the soil samples collected for the Dallas Lead Study was at first conducted on a "non-interfering" basis, however, since this study is of local concern, the soil sample analyses became later a high-priority item.

In 1972-73, the EQ Laboratory was involved in a study designed to obtain information on lead contamination resulting from lead smelter emissions and automobile exhausts. For this study, soil and other samples were collected and analyzed for lead. Since then the EQ Laboratory only infrequently analyzed soil and sludge samples, as requested by other departments, usually for Ca, Fe, Pb, Cd, Zn and other elements.

At the beginning of the study, the core samples (3" by 3/4" collected with a steel cork bore) were subdivided in the field in 1" segments (designated A, B, and C) which were analyzed separately in the EQ Laboratory. Later, after about 50 samples had been analyzed, only the top 1" segment of each sample was bagged in the field and analyzed in the laboratory since it was assumed that the main contamination risk for humans results from the top one-inch portion of the soil. The soil samples prepared for analysis usually contained some vegetation fragments.

This evaluation is restricted to items pertaining to the analysis of soil samples for lead. A copy of a Laboraory Evaluation Checklist used during the evaluation is enclosed as Appendix 2.

The evaluation team wishes to acknowledge the excellent cooperation provided by Mr. Rogan and his staff during this laboratory evaluation.

PERSONNEL: Until 1/20/82, Al Harris, now retired, was Laboratory Supervisor; since 1/20/82, Gary Rogan is Acting Laboratory Supervisor. Both have been working in this laboratory for more than ten years. They have both participated in earlier studies which involved lead determinations in soil samples. The persons involved in the extraction of the samples had one year and five months experience, respectively, and the AA spectroscopists, two years and two years and five months, respectively. Copies of their resumes are included as Appendix 3. The laboratory peronnel received on-the-job training by the senior chemists.

At the beginning of the study it was found during a routine data evaluation by the supervisor that one chemist had generated unacceptable data, as evidenced by extremely high and low values and by bad reproducibility of duplicate analyses. It was also found that the lead standard solutions prepared by this chemist were erroneous. Therefore, samples analyzed up.to the time of the discovery of the problem were reanalyzed. The chemist in questions is no longer with the laboratory.

A copy of the organization chart for the laboratory is enclosed as Appendix 4.

FACILITIES AND EQUIPMENT: During a tour of the EQ Laboratory it was verified that the laboratory facilities were adequate. There was sufficient laboratory and bench space, and a fume hood (which could be locked when the sample custodian was leaving the laboratory) was also available. Deionized water of adequate purity was available, as well as laboratory equipment required for the task (drying oven, desiccators, hot plates, shaker, glassware, class A volumetric flasks, etc.). The study was carried out following chain-of-custody guidelines. Examples of chain-of-custody record sheets are enclosed as Appendix 5. All samples and (unanalyzed) extracts were kept in secured areas. General housekeeping was excellent.

PROTOCOLS AND DOCUMENTATION: No separate written sample receipt, storage, sample extraction, and sample analysis operating procedures exist for this task, although such procedures for other types of samples are available (Appendix 6). A copy of the method used for the soil extraction and for the lead analysis is enclosed as Appendix 7. It is an adaptation of the method for lead determination on high-volume filters. Due to the close supervision exercised in this laboratory (as observed by the Evaluation Team), lack of written operating procedures for this task may not have been a problem during the sample preparation and analysis period.

A good, detailed general QA plan (dated 11/5/81) for the laboratory is available; its Table of Contents is enclosed as Appendix 8. However, this QA plan is tailored to air sampling and analysis which is in accordance with the mission of the laboratory as quoted from the QA plan: "The primary tasks of the Environmental Quality Laboratory are air sampling and analysis."

One individual is responsible for the EQ Laboratory quality assurance (Michael Sorenson - dual responsibility); another individual is responsible for sample control (Valerie Cooper).

The EQ Laboratory currently participates in the semi-annual Air Survey Interlaboratory Study conducted by RTP. This involves analyzing air filters that have been impregnated with lead.

Chain-of-custody sheets remained with the samples; whenever some of the sample was removed it was recorded on the sheet. The data generated were recorded in looseleaf notebooks. All data generated for a particular batch of samples (30-40 samples) were reviewed when the analysis of all samples of that particular batch was completed. Suspect results (e.g., duplicates that differed by more than ±7 percent) were flagged and the sample reanalyzed. The notebooks were reviewed only to follow up on suspect samples.

No bound maintenance logbook existed for the AAS. All calibration data, service activities (like lamp change) and other performance information pertaining to the AAS were recorded directly on the strip chart using a rubber stamp.

Contamination Control: All glassware used for this task for soil sample analysis was washed in a laboratory detergent and rinsed with deionized water.

One reagent blank was analyzed during each run (30-40 samples). According to the records these blanks revealed that no detectable lead was present in the reagent used (0.45 M HNO_3). No preparation blanks were analyzed.

The chemicals used for standards preparation were ACS Reagent Grade quality. Deionized water was used for all reagent and standard preparations. Standards were dated starting January 1982. The Texas Air Control Board recommendations were used to determine the shelf-lives of the standards.

Atomic Absorption Calibration and Methodology: An IL Model 551 Atomic Absorption Spectrophotometer with automatic background correction was used in the flame mode for the lead-in-soil determinations. The wavelength used was 283.3 nm.

The AAS was calibrated using working standards prepared daily from a stock solution (1000 ug Pb/ul). The stock solution was prepared from Fisher ACS Reagent Grade lead nitrate that had been dried for four hours at 120°C and cooled in a desiccator prior to weighing. The working standards were prepared in 0.45 M HNO_3 at concentrations up to 40 ppm Pb; the AAS was calibrated and used only in the linear range (up to 15 ppm Pb). Whenever the lead value of a sample extract was higher than 15 ppm, the sample extract was diluted to a concentration below 15 ppm and reanalyzed.

The AAS calibration was reevaluated by analyzing a calibration standard after every tenth sample. A ±5 percent drift required recalibration and reanalysis of the last ten samples.

Duplicate samples were analyzed at a rate of one duplicate per batch (30-40 samples). The duplicates were not "blind" to the analyst. The criterion was agreement within ±7 percent. If a set of duplicates failed to meet this criterion, the duplicate analysis was repeated with fresh material from the same soil sample.

At the beginning of this study, a soil residue from an extraction was reextracted and the extract analyzed for Pb. It was found that approximately 97 percent of the lead extracted during the two extraction steps has been removed during the first extraction (Appendix 9). Based on this result it was determined that one extraction per sample was sufficient for this study. However, since the chemist who reported these results was later found to have generated "bad" data (and was relieved of his responsibilities), this result is suspect.

No determinations of total lead in soil were performed. No other laboratory analyzed splits of the soil samples or the extracts. The extraction technique was not evaluated on NBS or other certified samples.

Discussion: The EQ Laboratory does not have an impressive background of lead-in-soil determinations, however, personnel qualifications, facilities and equipment of the EQ Laboratory seem to be adequate for the analysis of soil samples for lead. The fact that early in the study bad data were spotted and that corrective action was taken immediately demonstrated good supervisory practices. However, the lack of written sample receipt, storage, sample extraction and sample analysis operating procedures must be rated as a deficiency.

The protocol, as available (Appendix 7), must be judged as insufficient. The execution of the chain-of-custody procedures as used during this study was excellent.

No detailed QA plan had been prepared for this task. This must be rated as a deficiency. During the preparation and careful review of such a QA plan any deviations from good QA practices would most certainly have been apparent.

The contamination control procedures used in the sample preparation and analysis phase of this task were not quite adequate. It is a generally accepted practice to soak glassware in a nitric acid bath for a minimum of four hours, followed by a rinse with deionized water and air drying. Although the lead levels encountered in the soil samples are quite high it is still important to insure that the lead is completely removed from the glassware used. This is especially true for volumetric flasks since they are used to prepare standards and extract dilutions.

Only reagent blanks were analyzed. However, in order to determine the extent of contamination caused by reagents, glassware, and laboratory dust, laboratory blanks should be processed together with the samples at a minimum frequency of one blank per 20 samples. This will allow the analyst to detect contamination problems early thus enabling him to correct the problem.

Samples weighed in the balance room were left uncovered until extracted. Good laboratory practices include keeping beakers covered with watch glasses to avoid possible airborne contamination.

The lead nitrate used for the preparation of the AAS calibration standards (Fisher ACS Reagent Grade) was not dated and appeared to be aged. Standard solutions freshly prepared from this material should be compared with standard solutions freshly prepared from NBS-certified solutions used for calibration of the AAS. This would provide NBS traceability for the standard solutions used for the calibration of the AAS. It is important for laboratories participating in studies such as the Dallas Lead Study that traceability to some nationally acknowledged standard - preferably a NBS standard - is estabhished and maintained. The EQ Laboratory should routinely use the NBS-certified lead nitrate for all AAS calibrations, verify calibration accuracy by analyzing an independent standard after each calibration and after every 20 samples (EPA Quality Assurance samples could be used), maintain a control chart with these results, and calculate the 2 sigma limits to define when a corrective action, such as recalibration, is needed.

The EQ Laboratory is participating in an interlaboratory study on lead and air filters. Samples are analyzed twice a year. EQ Laboratory results for the filter samples analyzed in February 1981 showed a very slight low bias, whereas the results from the July 1981 samples showed a definite negative bias (from -0.5 percent to -17 percent). However, the July 1981 samples were analyzed by the analyst who was later shown to have generated questionable data and who is no longer with the EQ Laboratory. No results were available from the analysis of the February 1982 filter samples.

The efficiency of the soil extraction procedure, i.e., how much of the extractable lead was extracted during the one-step extraction procedure with nitric acid, has not been established. One attempt had been made to determine the extraction efficiency by reextracting a soil residue from an extraction, however, this should be done on a representative number of samples.

Total lead determination is not a requirement for the Dallas Lead Study, however, determination of the total lead content of approximately 10 percent of the samples collected from each geographical sampling area would allow determination of the percentage of total lead extract[illegible] Variations of this percentage for different areas would indicate either m[illegible] changes or changes of the chemical forms of the deposited lead, or a combination of both.

The precision of the sample extraction has been determined by extracting approximately 2 to 3 percent of the samples in duplicate. A more adequate number of samples that should be extracted in duplicate is 10 to 15 percent.

A method to determine the precision of the extraction procedure would be to extract NBS Standard Reference Material 1645, River Sediment, which should be analyzed as a control sample at a frequency of approximately 5 percent.

The accuracy achieved for the analysis of the soil extracts can not be determined from the data available. The method of analysis used does not allow a direct evaluation of the effect of the sample matrix (extractant plus soil extract) on lead absorption in the flame. The matrix effect could be positive or negative, depending on the nature of the matrix. The impact of the matrix effect on observed AAS results is commonly evaluated using the method of standard addition. When the results from the method of addition agree within ±10 percent with results using the working calibration curve it can be assumed that the matrix effect is negligible.

The composition of soil samples from different sampling locations may differ. This would impact on extractability of lead and on matrix effects. According to Mr. Ray Lozano, the soil is fairly uniform throughout the Dallas area. It is characterized as an elastic-type clay with large expansion and contraction properties, sometimes referred to as "alkaline gumbo-type clay." The uniformity of soil, if confirmed, would provide a justification for the use of single extraction for all soil samples collected, since the matrix effect would then be expected to be uniform. Dr. J. B. Cosley, Assistant Professor, University of Texas, Arlington, a geologist who is very experienced in soil characterization, has evaluated some of the soil samples which were collected and analyzed for the Dallas Lead Study by the University of Texas, Arlington team. She classified all samples she had evaluated thus far as silty clay/clayey silt, with some organic material. However, it was noted during the evaluation of the EQ Laboratory that the color of dried samples varied from dark to light gray possibly indicating a difference in composition.

Conclusions: Based on the quality control employed by the Environmental Quality Laboratory it is not possible to state the quality of the data generated. The data can be used for the detection of relative differences in extractable lead from samples collected at different sampling sites, however, no exact absolute value (with a defined accuracy) for extractable lead can be assigned to any one sample. Additional limited studies (detailed below) would provide more information on the accuracy of the data.

Recommendations: The following actions were recommended to Mr. Rogan of EQL during the debriefing session on March 18, 1982:

1. Prepare and use detailed protocols for sample receiving, sample storage, sample extraction, extract analysis, cleaning of glassware. Specify in the latter protocol that cleaning of the glassware with detergent and water should be followed by soaking the glassware in a nitric acid bath for a minimum of 4 hours, followed by rinsing with deionized water and air drying.

2. Provide the evaluation team with the February 1982 results from the interlaboratory study (lead on air filters). Acceptable results in this study would link the EQL AAS calibration to a national study.

3. Prepare standards from NBS-certified $Pb(NO_3)_2$ and compare them with standards prepared from the Fisher ACS Reagent Grade $Pb(NO_3)_2$ which was used in the past for the AAS calibration. This would provide NBS-traceability for the data previously generated. Use from now on only NBS-certified $Pb(NO_3)_2$ for all AAS calibrations.

4. Analyze from now on an independent standard (EPA quality assurance samples) after each calibration and during analysis at a rate of 5% of the samples analyzed. Maintain control chart with these results and plot the 2-sigma limits.

5. Select approximately 50 samples, if available, from those previously analyzed for this task which should be representative of the areas, the lead concentration levels (low, medium, high), and color and consistency of the soil samples. It would be desirable to include in this selection of samples as many of those samples that had previously been analyzed in duplicate. Send those samples to Dr. Tom Spittler, Region 1, who has agreed to analyze them for total lead content using x-ray fluoroscence techniques.

6. Select approximately 10 samples from each area (which encompass low, medium, and high concentrations of lead), spike these samples with $Pb(NO_3)_2$ at concentrations similar to those of the extractable lead determined earlier for these samples, extract the spiked samples and analyze the extracts for lead

using the method of standard addition. If the recovery of the lead spikes is 85 to 115 percent this could demonstrate the method's ability to extract known amounts of lead from the samples. If possible the samples should be selected from those selected under item 5 above.

7. Determine the precision of the extraction method by extracting several samples of NBS SRM 1645 - River Sediment - and analyzing the extracts for Pb. During routine analysis, such an extraction should be performed after every set of approximately 20 samples.

8. Verify the extraction efficiency by repeated extraction of several samples per area selected according to the criteria detailed in item 5 above. Preferably these samples should be selected from those to be analyzed for total lead.

9. Apply the method of standard addition to the extracts of approximately 10 percent of the samples selected according to the criteria in item 5 above and compare the results with the working calibration graphs. An agreement within ±10 percent would indicate that there is no appreciable matrix effect which would bias the AAS data.

10. Analyze samples in duplicate at a 10 percent frequency; the duplicates should be blind to the analysts. Close agreement of these duplicate results would indicate a high degree of sample homogeneity and precision of the extraction method. If possible, those samples selected under item 5 above that had not been analyzed in duplicate should be analyzed now as duplicates.

11. Prepare and put immediatly into effect a detailed QA plan which includes all the pertaining items recommended here.

Werner F. Beckert
Werner F. Beckert, Ph.D.
Program Manager
Quality Assurance Division

Stephen C. Simon
Steve Simon, Ph.D.
Program Manager
LEMSCO

BEST COPY AVAILABLE
FOR CLARIFICATION CONTACT
KENNETH W. BROWN (702) 798-2214

U.S. ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS
P.O. BOX 15027, LAS VEGAS, NEVADA 89114 · 702/736-2969 (FTS 545-2969)

Date:

Reply to Attn of: QAD

Subject: Evaluation of ESA Laboratories, Inc., Bedford, Massachusetts, March 23, 1982.

To: Files

Persons Contacted:	Dr. Reginald M. Griffin, Laboratory Director, ESAL Dr. Thomas R. Copeland, Technical Director, ESAL Ms. Jane Buono, Laboratory Manager, ESAL
Evaluation Team:	Dr. Werner F. Beckert, EMSL-LV Dr. Steve Simon, LEMSCO, Las Vegas
Evaluation Observers:	Dr. Thomas Spittler, Region 1 Dr. Jim Simpson, Center for Disease Control, Atlanta

Purpose: To evaluate ESA Laboratories, Inc., with respect to blood lead analyses performed for the Dallas Lead Study.

Background: Personnel from the City of Dallas Environmental Health and Conservation Department collected blood samples from people living in the vicinity of two active lead smelters in Dallas, Texas. These samples were sent to the ESA Laboratories and analyzed for lead using anodic stripping voltammetry (ASV). A total of 11,545 blood samples was analyzed for lead for this study between 9/24/82 and 11/25/82.

This evaluation is restricted to items pertaining to the analysis of Dallas blood samples by ESAL for lead. A copy of a Laboratory Evaluation Checklist used during the evaluation is enclosed as Appendix 1.

The evaluation team wishes to acknowledge the excellent cooperation provided by Dr. Griffin and his staff during this laboratory evaluation.

Laboratory Performance History: ESA Laboratories analyze a wide range of samples for metals and trace metals, including water and waste water samples, food stuffs, clinical specimens including blood, hazardous wastes (EPA "EP toxicity") and many others. ESAL offer erythrocyte protoporphyrin and zinc protoporphyrin analyses. Gas chromatographic analyses for common organic solvents are done on air, bulk or clinical samples. The facility is licensed by the Centers for Disease Control (CDC); the States of Connecticut and New York as a clinical laboratory; is registered with the Commonwealth of Pennsylvania as a clinical laboratory; is accredited by the American Industrial Hygiene Association and is certified by the Commonwealth of Massachusetts (for EPA) to perform water analysis. ESAL is a reference laboratory for the HHS-CDC (B.S.S.) blood lead and erythrocyte protoporphyrin proficiency testing program and for those sponsered by the States of Pennsylvania, California, New York and New Jersey. It has been approved by CDC for blood lead analysis (Appendix 2). For many years, the laboratory has routinely conducted blood analyses for lead and other parameters.

ESAL regularly participate in two different CDC Blood Lead Proficiency Testing Programs. Appendix 3 lists ESAL's 1981 performance in the first Blood Lead Proficiency Testing Program (3 samples quarterly). Successful participation in this program is part of the liscensing requirements for interstate laboratory commerce. It can be seen from the data sheet that ESAL's performance was acceptable for all of 1981. Appendix 4 lists the performance of ESAL in the second Blood Lead Proficiency Testing Program (3 samples monthly) for December, 1981. In this latter program (which is conducted for the National Lead Poisoning Prevention Program) ESAL participate twice since ESAL are one of the six reference laboratories (Code 1-001) and also one of the participating laboratories (Code 77). Again ESAL's performance was acceptable.

ESAL also participate regularly in the WS and WP interlaboratory studies conducted by the EMSL-CIN. The following results from two recent studies were obtained and verified using Region 1 records:

WS008	Pb Reported (ug/l) by ESAL	True Value (ug/l)	Acceptable (ug/l) Limits
(7/30/81)	83.9	80.5	63-99
	16.8	12.0	4.6-20
WP007			
(10/15/81)	225	210	167-252
	29	27.1	17.6-39.6

Personnel: A copy of the organizational chart for the laboratory is enclosed as Appendix 5.

Dr. Griffin, Dr. Copeland and Ms. Buono have each more than 10 years of laboratory experience; their resumes are enclosed as Appendix 6. The technicians who worked on this task have between 6 months and 3 years of laboratory experience.

Facilities and Equipment: During a tour of the ESA Laboratories it was verified that the laboratory facilities were adequate. There was sufficient laboratory refrigeration and bench space available for sample storage, preparation and analysis; a perchloric acid fume hood in good condition was also available. Deionized water of adequate purity was available as well as all laboratory equipment specified in the ESAL "Blood Lead Lab Procedures Manual", Section VIII (Appendix 7).

Protocols and Documentation: The "Blood Lead Lab Procedures Manual" (Appendix 7) includes protocols for sample receipt, sample log-in procedures, equipment decontamination procedures, preparation of reagent solutions and standard lead solutions, sample preparation, sample analysis, result calculations and reporting, equipment maintenance, laboratory safety, and personnel training policy. One individual (George Scott) is responsible for the labortory QA; another individual (Tom Cronin) is responsible for sample control.

No separate QA plan has been written for this task, but adequate QC is included in the individual protocols. During the laboratory tour the evaluation team verified familiarity with and adherance to this protocol by the laboratory personnel.

No chain of custody was required for this task.

The data generated for this task were recorded on special lab work sheets (Appendix 8). No single individual at ESAL is responsible for maintaining all records and documents. The Laboratory Supervisor reviews and transposes all data which are then reviewed by the Laboratory Director.

There is a logbook for each instrument for recording performance information such as calibration data and service activities.

All samples are kept refrigerated at 3°C; sample extracts are covered with Saran wrap when kept overnight. Storage time at ESAL for the blood samples received for the Dallas Lead Study did not exceed 4 days except for samples received during the week of October 13 when some samples were stored for 5 to 6 days.

Contamination Control: Glassware used for blood lead analysis is decontaminated by a 24-hour acid leach. The leaching solution is then analyzed for lead content and the acid leach repeated, if necessary, until the lead content of the leaching solution is equal to or smaller than the lead value in blanks.

Vacutainers used by ESAL customers for collecting blood samples are checked for lead contamination on a regular basis as described in Appendix 7, Part IV.

The reagents used in the blood-lead procedure are checked for lead contamination as described in Appendix 7, Part III. Control limits exist for acceptance or rejection of each material.

Procedure blanks are prepared at the rate of one per six samples.

ESAL's perchloric acid hood is washed down every Friday.

Instrument Calibration and Procedural Quality Control: The blood samples from the Dallas Lead Study were analyzed using an ESA Model 2014 Multicell Anodic Stripping Voltammetry system. The analyses cells are standardized twice daily with standards prepared from NBS-certified lead nitrate to determine the "cell correction factor" for each cell. Calibration for blood lead analysis is performed by the use of organic blood standards. These standards consist of samples of pooled blood of low endogenous lead value which are spiked with 20, 50, or 100 ug% Pb. Each spiked solution is analyzed in duplicate; one set of organic blood standards is analyzed with each set of samples (which consists of 152 or less samples).

A set of laboratory control standards is analyzed daily. These standards consist of blood composites at three lead levels: low (20-30 ug%), intermediate (50-60 ug%) and high (90-110 ug%). Each control standard is analyzed daily in duplicate and the results are plotted on a control chart. Corrective procedures are applied when any standard is outside of the acceptable range.

The efficiency of the digestion process is verified on each sample and blood standard by monitoring the lead peak shape and the copper peak adjacent to the lead peak. When the digestion is complete the copper peak goes off scale on a 0.2 m.a. full scale recorder. Should the copper peak not go off scale then incomplete digestion is assumed and the sample preparation and analysis are repeated.

Precision of the measurements is monitored through the RSDs of the inorganic and blood level standards. Acceptance criteria are well defined and well within criteria established by CDC.

Approximately 70% of the blood samples for the Dallas Lead Study were analyzed in duplicate. Of the remaining 30%, only 10% were analyzed in duplicate, due to a high sample load. This change was approved by the City of Dallas. When the results of any of the single-analysis samples showed a lead value greater than 25 ug%, these samples were reanalyzed in duplicate.

Discussion: Personnel qualifications, laboratory qualifications, facilities and equipment of the ESAL seem to be adequate. Documentation and documentation control were adequate, as evidenced by the detailed protocols in Appendix 7. No formal QA plan exists for lead-in-blood analysis, however, the QA incorporated into the protocols is adequate.

The procedures for contamination control used during the sample preparation and analysis phase were adequate for this task. Glassware cleaning procedures were monitored by analyzing cleaning solutions for lead, and the analysis for lead of all reagents used for this task assured minimum contamination by reagents. During a tour of the facilities it was noted that housekeeping was adequate and that good laboratory practices were followed.

Sample digestion efficiency is monitored by the copper peak. ESAL have shown in the past that this method is adequate to identify incompletely digested samples.

NBS-certified lead nitrate is used to prepare all standards and spikes. ESAL make their own blood standards, however, these standards are checked against standards made with NBS-certified lead nitrate.

The number of blanks, spiked samples and standards analyzed amounts to approximately 15% of the number of samples analyzed. In light of the vast experience of this laboratory in blood lead determination, and in considering their good performance in several interlaboratory comparison studies, this seems to be adequate.

Conclusions: The information collected during the evaluation of ESAL convinced the evaluators that ESA Laboratories are highly qualified for analyzing lead in blood. This is based on the following:

1. The performance history of the laboratory in national blood lead proficiency testing and certification programs.

2. The qualifications of the staff and the documented staff training program.

3. Documentation of procedures and control practices.

4. Quality of facilities and equipment.

5. Good laboratory practices which are encouraged by management and followed by the laboratory staff.

The evaluators found no indications that the analytical and QA/QC procedures used during the analysis of the blood samples from the Dallas Lead Study were any different from those documented in the protocol.

Werner F. Beckert
Werner F. Beckert, Ph.D.
Program Manager, QAD

Stephen J. Simon
Steve Simon, Ph.D.
Program Manager, LEMSCO

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Date: JUN 2 9 1982

Reply to Attn of: QAD

Subject: On-Site Laboratory Evaluation of GCA Corporation, Technology Division, Bedford, MA, June 23, 1982, for the Dallas Lead Study

To: Files

Evaluation Team:	Werner F. Beckert, EMSL-LV Stephen J. Simon, LEMSCO, LV
Persons Contacted:	Ken McGregor, Laboratory Director Deborah McGragh, Inorganic Section Head Tom Hibson, AA Analyst Mary Kozik, QA Officer Sandra Sandberg, Sample Bank Manager

Purpose: The purpose of this laboratory evaluation was to determine whether GCA, a potential contractor for the analysis of soil samples for lead, had facilities, personnel and experience adequate to carry out a large number of soil analyses for lead, as well as a thorough understanding of the Dallas Lead Study-Soil and Wipe Sample Analysis Protocol, Revision #1, including the detailed QA/QC features.

Protocol Discussion: The following parts of the Soil and Wipe Sample Analysis Protocol, Revision #1, were discussed in detail and modified:

1.1.2: GCA intends to use block digestors in addition to, or in place of, hot plates. Since block digestors actually improve digestion temperature control, 1.1.2 was amended to read: "1.1.2 Hot plates and/or block digestors..."

1.2.2: GCA routinely uses Baker Instra nitric acid which contains <.0002 ppm Pb. This is less than ACS Reagent Grade lead content which is specified as <.005 ppm. 1.2.2 was, therefore, amended to read: "1.2.2 Concentrated nitric acid (HNO_3), ACS Reagent Grade or better."

1.3: GCA personnel routinely clean their glassware by refluxing it with 1:1 HNO_3 or, for volumetric flasks, by soaking them in 1:1 HNO_3. Since in the opinion of the evaluation team the GCA cleaning method is more rigorous than that specified in the protocol, the first part of 1.3 was amended to read: "1.3 All laboratory ware used for extraction, analysis and storage will be cleaned in the following manner (unless a variation equaling or exceeding the following method has been approved by the evaluation team)."

1.7.3: The second sentence was amended to read: "Take the mean of three integrations per sample (integration time: 5 to 7 seconds) and record this value..."

1.8.1: The evaluators agreed with GCA analysts that the minimum reportable level of 4 ug Pb/gm and the detection limit for lead of 0.1 ug Pb/ml were set unnecessarily low for these kind of samples. This part of 1.8.1 was therefore changed to read: "The minimum reportable level is 8 ug Pb/gm based on twice an instrumental detection limit for lead of 0.2 ug Pb/ml."

2.1.5: In the protocol, it is not specified which sample extract should be spiked with lead nitrate. The following statement was therefore added to the first paragraph of 2.1.5: "The parent soil sample will be sample x_6."

2.1.5.b: It was clarified that the amount of lead nitrate to be added is (1) determined on the basis of the final dilution, if applicable, and (2) added to this dilution. The first sentence of 2.1.5.b was therefore changed to read: "b. To an aliquot of this sample extract or its final dilution, if applicable, is added an amount of lead (as lead nitrate) approximately equal to the lead content determined for this extract or its final dilution, if applicable, but greater than 10 x the instrumental detection limit."

2.1.6.a: Add: "The parent sample will be x_{12}."

2.1.7: Add after the first sentence: "The parent samples will be x_9 for g_1 and x_{20} for g_2."

2.1.8.b: Was clarified to read: "The 5-gram samples are to be prepared and analyzed as detailed in Appendix C and in Section 1.6.1.b through f (except for the grinding and sieving) and 1.7."

2.1.9.a: Was changed to read: "For each 20 soil sample extracts, split the extract from sample x_8 (see page 19) with two portions, analyze the first portion as extract x_8, the second portion as i following x_{18}."

2.1.10.a: Was changed to read: "For each 20 samples, a duplicate portion of sample is spiked with 700 ug Pb as lead nitrate solution per gram of sample, and processed as specified in 1.6 and 1.7. EPA may request a change of the amount of lead added during the course of the study."

In addition the following items were discussed:

1. The reporting forms included in the protocol as Figures 1 to 4 are not the final form. Reporting requirements (frequency and recipients) have not been finalized yet.

2. Dr. McGregor was concerned about the chain of command within EPA: who will direct his efforts, who will decide on the payments to the contractor and on what basis?

3. It is possible that a number of "total recoverable" analyses will be carried out at the beginning of the analytical program. This could mean that the 2.1.7 requirement might be eliminated.

Equipment and Space: GCA possesses the equipment required for this task. This includes a Perkin-Elmer 460 Atomic Absorption Spectrophotometer with background corrector and, as a back-up AA system, a Varian AA6 with deuterium lamp background corrector. Downtime for their instruments has generally been less than 10 percent; in case of instrumental problems they usually get service within 24 hours. GCA is at present and for the forseeable future not committed to any projects that would require substantial AA time.

Up to 10 hoods will be made available for this program, as needed.

The bench space available for this program is adequate.

Documentation and Chain of Custody: GCA has SOPs for laboratory operations available, as well as adequate QA plans. Their chain-of-custody documentation is adequate. Logbooks are being used for instrumentation and balances; balances are routinely checked with Class S weights. All operators use notebooks routinely; the notebook entries are checked and co-signed by the supervisor whenever an assignment has been completed. The QA Officer periodically audits the notebooks.

The GCA Sample Bank Manager assigns GCA sample numbers to all samples received. Every single number that has been transcribed during the course of a project is either verified by the QA Officer or by the Inorganic Section Head.

Enough secured ambient-temperature sample storage area is available for this project. GCA will provide additional secured storage space for sample in preparation, including locked hoods and cabinets.

Personnel: The following individuals will be assigned to this program:

Deborah McGragh, Program Manager
Sandra Sandberg, Sample Branch Manager
Mary Kozik, QA Officer
Tom Hibson, AA Analyst
Ho Ling Yee, Sample Preparation Specialist
Sean Morris and Allan Peterson, Grinding, Sieving and Weighing

Alternates are Andrea Cuttey and Barbara Myatt; both are qualified and available to perform AA and/or sample preparation.

Resumes of the key employees listed above are enclosed.

Additional personnel from within the Division will be made available, if needed. GCA would probably use a 12-hour shift for this program.

Laboratory Walkthrough: During a laboratory walkthrough, the use of logbooks, SOPs, etc., the availability of instrumentation and labware and efficiency of the chain-of-custody and sample handling procedures were verified. Housekeeping was adequate in the laboratories to be used for this project. Potential sample flow from receipt of samples through preparation, analysis, data reporting, validation and sample storage was deemed adequate to meet the projected needs of this program.

Conclusions: The evaluators feel, based on the above facts, that the GCA Corporation, Technology Division Laboratory, has adequate facilities and equipment, as well as adequate experienced personnel available to analyze up to approximately 150 soil samples for lead per day, assuming a 12-hours shift.

for Donald J. [illegible]
Werner F. Beckert, Ph.D.
Program Manager
Methods Improvement & Testing Program
Quality Assurance Division

S. Simon
Stephen Simon
Principal Scientist
Environmental Programs
LEMSCO

U.S. ENVIRONMENTAL PROTECTION AGENCY
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Date: JUN 29 1982

Reply to Attn of: QAD

Subject: On-Site Laboratory Evaluation of Envirodyne Engineers, Analytical Services, St. Louis, MO, June 24, 1982, for the Dallas Lead Study

To: FILES

Evaluation Team: Werner F. Beckert, EMSL-LV
Stephen J. Simon, LEMSCO-LV

Persons Contacted: Thomas H. Lachajczyk, Senior Environmental Scientist
Carol H. Byington, Manager, Analytical Services
Cliff J. Kirchmer, QA Officer
Judy Stone, Sample Bank and Data Manager
Paul B. Humburg, AA Analyst
Paul R. Myers, Sample Preparation Manager

Purpose: The purpose of this laboratory evaluation was to determine whether Envirodyne, a potential contractor for the analysis of soil samples for lead, had facilities, personnel and experience adequate to carry out a large number of soil analyses for lead, as well as a thorough understanding of the Dallas Lead Study--Soil and Wipe Sample Analysis Protocol, Revision 1, including the detailed QA/QC features.

Protocol Discussion: The protocol was discussed in detail, and all changes of Revision 1 that had been effected on our master copy of the protocol were conveyed to Envirodyne personnel. The following parts of the protocol were modified or amended:

1.3: The evaluators agreed that the Envirodyne's routine method for glassware cleaning by refluxing with 1:1 nitric acid is adequate.

1.5.2.a. and b: Their instrument microprocessor can accept a maximum of 5 calibration standard points. "6 calibration standards" was therefore changed to read "5 calibration standards."

2.1.1.c: Change "separative" to "repetitive."

2.1.2.d: Change "outside the 95% confidence limits" to read "outside the 99% confidence limits."

2.1.8.e: Change "95% confidence limits," to read "99% confidence limits."

2.1.10.d: Change "95% confidence limits," to read "99% confidence limits."

In addition, the following items were discussed:

1. The reporting forms included in the protocol as Figures 1 to 4 are not the final forms. Reporting requirements (frequency and recipients) have not been finalized yet.

2. It is possible that a number of "total recoverable" analyses will be carried out at the beginning of the analytical program; this could mean that the 2.1.7 requirement might be eliminated.

3. Envirodyne personnel would like to have a list of people who would be authorized to directly request data from Envirodyne. Further, they would like to know to whom to report any sample receiving problems (e.g., broken chain-of-custody seal, split sample bags, etc.).

Equipment and Space: Envirodyne possesses now, or will acquire as needed (1-week delivery for lab ware), the equipment required for this task. The available equipment includes an Instrumental Laboratory 251 Atomic Absorption Spectrophotometer with background corrector and automated sampler and, as a backup system, an identical second unit. Downtime of these instruments has generally been <10 percent. They have a service contract in force (response time usually 24 hours) but perform many repairs themselves. Envirodyne has continuing AA commitments, mainly for DOD projects; they will therefore conduct this program using a second shift. Up to five hoods will be made available for this purpose, as needed. The bench space available for this program is adequate.

Documentation and Chain-of-Custody: Envirodyne has a good laboratory QA plan available, as well as SOPs for instrument calibration. Logbooks are being used for instrumentation use but not yet for instrumentation maintenance. Laboratory notebooks are used routinely; data sheets are taped into notebooks, as needed. Notebooks entries and data sheets are reviewed by the supervisor when sets of measurements have been completed.

The QA plan contains a short, general section on sample receipt. Specific receipt and sample handling SOPs are being prepared for this program. All samples received are assigned Envirodyne sample numbers.

Envirodyne has handled contracts that required chain-of-custody documentation. The chain-of-custody requirements for the soil sample analysis for the Dallas Lead Study were discussed in great detail with Envirodyne personnel who will handle soil samples. Secured areas have been identified where the samples will be stored when received and during the various processing steps. Dr. Kirchmer, the QA Officer, will assist the Sample Bank and Data Manager in implementing the chain-of-custody requirements for this program.

All numbers generated during the soil analysis for the Dallas Lead Study that have to be transcribed will be checked for transcription errors by the Sample Bank and Data Manager.

Personnel: The following individuals will be assigned to this program:

Carol Byington, Program Manager
Judy Stone, Sample Bank and Data Manager
Cliff Kirchmer, QA Officer
Alvin Byington, AA Analyst
Paul Humburg, Alternate AA Analyst
Paul Myers, Sample Preparation Manager
Several technicians, as needed, for grinding,
  sieving and sample preparation

Resumes of the key employees listed above are attached.

Laboratory Walkthrough: During a laboratory walkthrough, use of log books, SOPs, etc., the availability of instrumentation and lab ware, and the efficiency of the chain-of-custody and sample handling procedures were evaluated. Housekeeping was adequate in the laboratories to be used for this program. Suggestions were made concerning the potential sample flow from receipt of samples through preparation and analysis, sample storage and chain-of-custody procedures.

Conclusion: The evaluators feel, based on the above facts, that the Envirodyne Engineers, Analytical Services Laboratory, has adequate facilities and equipment as well as adequate experienced personnel available to analyze up to approximately 150 soil samples per day. Several specific suggestions made by the evaluation team will, when implemented, satisfy the chain-of-custody requirements for this program.

S. Simon

Stephen J. Simon
Principal Scientist
Environmental Programs
LEMSCO

Don Gurka

Werner F. Beckert
Program Manager
Methods Improvement & Testing Program
Quality Assurance Division

Lockheed Engineering and Management
Services Company, Inc.

Environmental Programs Office
944 E. Harmon Avenue, P. O. Box 15027
Las Vegas, Nevada 89114
(702) 798-2175

June 28, 1982

U. S. Environmental Protection Agency
P. O. Box 15027
Las Vegas, Nevada 89114

Attention: P. Richitt

Subject: Pre-Award Sample Results - Dallas Lead Study

Dear Mr. Richitt:

Three samples, each in duplicate, were sent on 6/18/82 to the two contractor laboratories being evaluated for the soil analytical work for the Dallas Lead Smelter Study. The laboratories involved were:

GCA Corp., Technology Division, Bedford, MA

Envirodyne Engineers, St. Louis, MO.

One set of samples were "blank" soils from the Las Vegas area having a lead content ≤ 15 ug/gm.

One set of samples were spikes on the above having a lead content of approximately 215 ug/gm.

One set of samples were NBS SRM 1648, River Sediment having a lead content of 714 ± 28 ug/gm.

Each laboratory received an updated (Revision 1) protocol and an instruction page on how to precess the samples.

Results:

Laboratory/Sample	BLANK	SPIKED SOIL	NBS SRM 1648
EMSL-LV	12 ug/gm ± 1	214 ug/gm ± 21	715 ug/gm ± 23
(# of determinations)	(8)	(7)	(8)
Acceptance Criteria	-----	171-256	669-761
GCA	4 ± 0.0	200 ± 1	684 ± 3
Envirodyne	12.5 ± 0.3	204 ± 8	682 ± 20

Conclusions:

Both laboratories were within the acceptance criteria established from EMSL-LV data on the spiked soil and NBS 1648. The acceptance criteria were established at ± 2 standard deviations from the mean value determined at EMSL-LV using the draft protocol. This criteria was believed to be reasonable based on the QC limits agreed upon (typically a control and warning limit of 3S and 2S respectively) and the overall QA requirements of this project.

GCA reported results slightly lower than determined by the other two laboratories on the "blank" soil and Envirodyne had one of the two River Sediment results on the low end (661 ug/gm); however, the mean result was within the acceptance window. In general, both laboratories evaluated performed on a roughly equal basis in this evaluation.

Very truly yours,

S. Simon

Stephen J. Simon
Task Leader

SJS/eg

cc: W. F. Beckert
R. K. Robeson
W. D. Reynolds
J. O. 74.21

APPENDIX D

QUALITY ASSURANCE PLAN
DALLAS LEAD STUDY

DA 68-02-3168

July 1, 1982

QUALITY ASSURANCE PROJECT PLAN

FOR

QUALITY ASSURANCE PLAN
DALLAS LEAD STUDY
DA 68-02-3168

XX New

____ Continuing

____ Revised

Environmental Monitoring Systems Laboratory
P.O. Box 15027
Las Vegas, NV 89114

APPROVAL: Kenneth W. Brown 7/6/82 Project Officer
Signature Date

7/6/82 Division Director
Signature Date

John A. Santolucito 7/6/82 QA Officer
Signature Date

Quality Assurance Plan Revisions

Date revised - September 1, 1982

1. Section 1.0 page 5 of 5. Referee Laboratory. Change EPA NEIC and Dr. Ted Meiggs to EPA EMSL-LV, Dr. Steve Simon.

2. Section 2.0 page 4 of 7. Change NEIC Denver to EMSL-LV, Las Vegas.

3. Section 2.0 page 5 of 7. 2.4 QC sample analysis. Omit NEIC Denver.

Date revised - August 27, 1982

1. Section 7.0. See Attachment 3 Soil and Dust Analytical Protocol for revisions.

Date revised - June 20, 1982

1. Section 9 page 7 of 10, output data. Raw data files will not be sent to CDC for their use.

Date revised - October 18, 1982

1. Section 3.0 page 1 of 3 (3.2 Precision and Accuracy). Change first sentence to read: The QA objective is to achieve an intralaboratory precision (for the analysis only) of ±10% with a recovery of lead of 90 to 110% as determined from the calibration check standards.

   Second sentence (clarification) the split samples referred to are the two soil samples shipped to the same analytical laboratory. See Soil Sampling and Sample Bank Procedures Protocol page 6 of 13, last paragraph, page 6 of 14 first paragraph, and Attachment 2 Sample Bank Procedures page 9 of 9 (7) last sentence.

2. Section T of C page 2 of 3 (Attachment Number 1). Change Soil Sampling Protocol to Soil Sampling and Sample Bank Procedures Protocol.

QUALITY ASSURANCE PLAN
DALLAS LEAD STUDY

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2. Dust Sampling Protocol
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4. Proposed Survey Design for Investigating the Effect of Traffic and Smelter Lead Emissions in Dallas, Texas

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5. Chain-of-Custody Record Sheet
6. Chain-of-Custody Record Sheet
7. Chain-of-Custody Record Sheet
8. Data Flow Diagram
9. Corrective Action Request Form

Appendix

1. Suggested Format for Analytical Subcontractors' Quality Assurance Plans.

Quality Assurance Plan
Dallas, Texas Lead Study

1.0 PROJECT DESCRIPTION AND ORGANIZATION

Data collected by the city of Dallas and the University of Texas at Arlington, have shown some soils collected in the immediate vicinity of the Dixie Metal Company (DMC) and the RSR Corporation (RSR) have lead levels that exceed 1,000 µg/g. In addition, blood samples collected from a number of individuals living in the vicinity of these two secondary lead smelters have contained lead levels that exceed 30 µg/dl.

The U.S. Environmental Protection Agency (EPA), the U.S. Public Health Service's Center for Disease Control (CDC) and the City of Dallas have designed an environmental multimedia monitoring study that includes the analysis of soils, house dust, paint and blood samples collected near these two smelter sites. As two major sources of lead are suspected; one from the smelters and one from automobile emissions, a third geographical area identified as a Reference Area (REF) will be selected for sampling. The selection of a REF site wll permit an assessment of the smelter lead contribution, as the REF sitewill contain as near as possible all physical and biological paramters exhibited at the RSR and DMC sites except for the smelter emissins.

The data obtained from this monitoring effort will be used to correlate blood lead levels with selected environmental media. Also, the data will be used to identify geographical areas that may require a remedial response.

Soils located in the immediate vicinity of homes, at parks, playgrounds, and schools will be collected. In addition, soils at designated sampling grid locations will be collected.

The blood sampling program includes the collection of blood from pre-school aged children. This effort will be complemented by the collection of house dust samples and the measurement of lead in paint from the homes in which the sampled children reside.

This Quality Assurance Plan includes the following attachments: 1) soil sampling protocol, 2) dust sampling protocol, 3) soil and dust analytical protocol, and 4) proposed survey design for investigating the effect of traffic and smelter lead emissions in Dallas, Texas.

1.1 Project Organization and Responsibility

Research Triangle Institute (RTI) is under contract to the U.S. EPA to provide the study design; Joseph Carra, Chief of EPA's Design and Development Branch, Washington, D.C., is the EPA Project Officer. Robert M. Lucas is the RTI Project Officer.

The City of Dallas will conduct the sampling effort in conjunction with EPA Region VI. The City of Dallas will also provide the analysis for all dust samples. Soil analyses will be provided by a separate contractor or contractors.

The U.S. EPA's Environmental Monitoring Systems Laboratory at Las Vegas (EMSL-LV) will provide the soil sampling protocol and the analytical protocol dealing with the analysis of dust and soils. EMSL-LV will provide the Quality Assurance Plan and will provide necessary information and direction to analytical laboratories and sampling teams such as: training, performance audits, internal quality control checks, laboratory evaluations, and the sample bank and chain-of-custody procedures. In addition, EMSL-LV will validate all dust and soil data, provide analytical support as a referee laboratory for up to 100 samples, if the National Enforcement Investigations Center (NEIC) becomes overloaded as principal referee laboratory and provide to the EPA Project Officer a report describing and identifying soil lead concentrations collected from grid sampling locations in the vicinity of the RSR, DMC and REF study sites.

The CDC will provide the paint and dust sampling protocols and the blood sampling procedures. The CDC will also be responsible for training sampling teams, selecting all soil sampling locations, excluding soil grid locations, and providing to the EPA Project

Officer a report describing and assessing existing correlations of blood lead levels with environmental lead levels in soils, dust, and paint. The analytical and quality assurance protocols and procedures dealing with blood will be provided by CDC. The CDC will also provide the required quality assurance audits and evaluations of the sampling teams and blood analytical laboratories.

The U.S. EPA in Dallas will provide the On-Scene Coordinator, oversee the sample bank operation, provide necessary training, audit and evaluate the sampling teams. The U.S. EPA's National Enforcement Investigations Center in Denver (NEIC) will provide analytical services as a referee laboratory and provide guidance for analytical and chain-of-custody methods and procedures.

Figure 1 shows the organizational chart for the Dallas Lead Study.

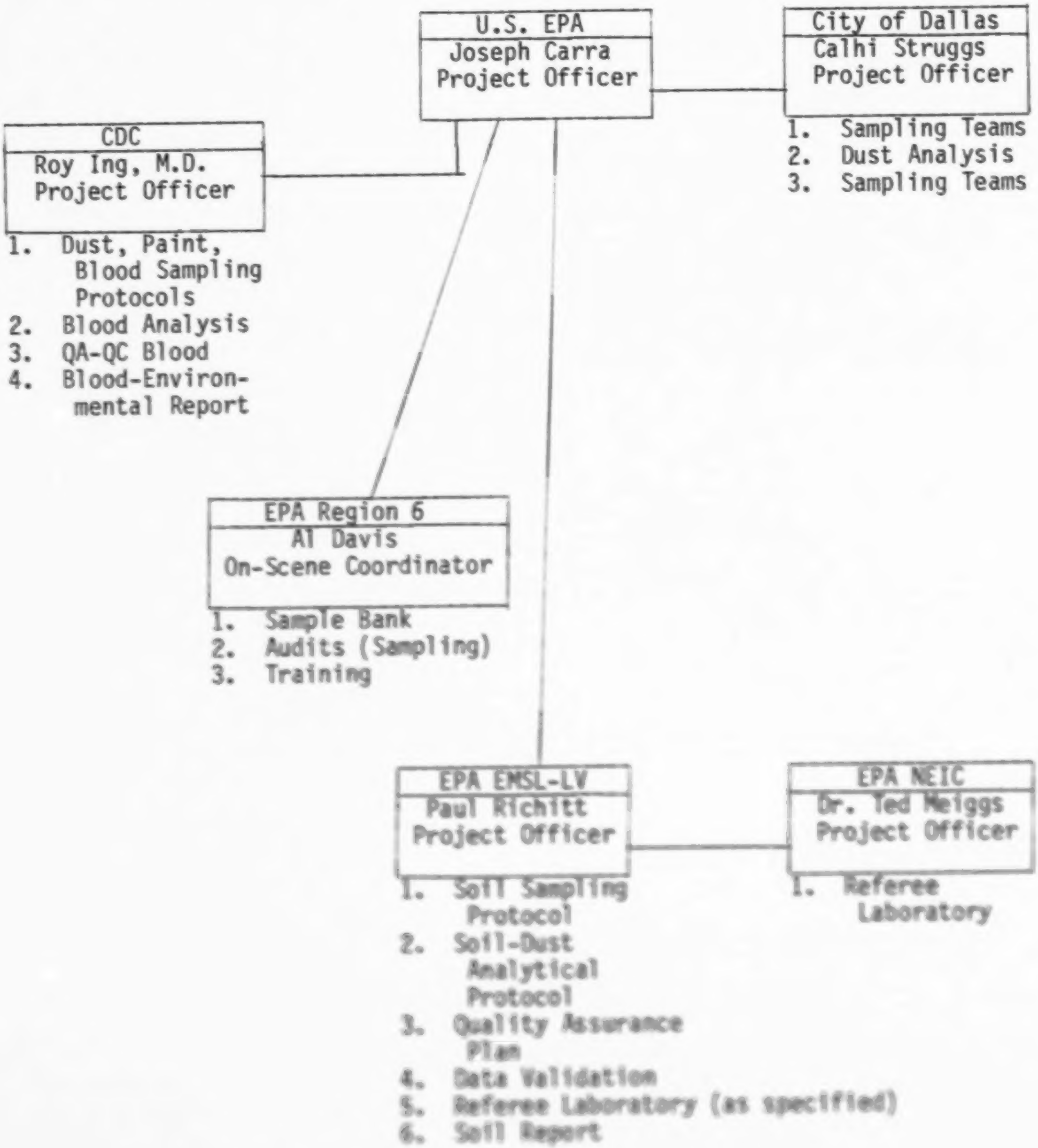

U.S. EPA
Joseph Carra
Project Officer
City of Dallas
Calhi Struggs
Project Officer
1. Sampling Teams
2. Dust Analysis
3. Sampling Teams
CDC
Roy Ing, M.D.
Project Officer
1. Dust, Paint, Blood Sampling Protocols
2. Blood Analysis
3. QA-QC Blood
4. Blood-Environmental Report
EPA Region 6
Al Davis
On-Scene Coordinator
1. Sample Bank
2. Audits (Sampling)
3. Training
EPA EMSL-LV
Paul Richitt
Project Officer
1. Soil Sampling Protocol
2. Soil-Dust Analytical Protocol
3. Quality Assurance Plan
4. Data Validation
5. Referee Laboratory (as specified)
6. Soil Report
EPA NEIC
Dr. Ted Meiggs
Project Officer
1. Referee Laboratory

Figure 1. Dallas Lead Study Organization and Responsibility.

2.0 MAJOR PROGRAM ELEMENTS

2.1 Sampling Plan

The study sites include all the area within a 1-mile radius of the RSR Corporation's smelter located near the intersections of Singleton Blvd. and Westmorland Avenue, and all the area lying within a 1-mile radius of the DMC smelter located near the intersections of McGowan Street and Sargent Roads in Dallas, Texas. A third study site having the identical configuration and area as the RSR and DMC study sites will be selected and identified as a reference site.

EPA statisticians will check the sampling protocols to ensure a statistically valid number of samples were collected. Soils, dust, and paint will be analyzed to provide lead data that may be correlatable to blood lead levels. Additional soils will be collected and analyzed to provide data that will be used to identify concentration isopleths for the three areas.

Aerial photographs will be provided for each of the three study sites. A grid measuring 750' x 750' will be overlaid on the photographs identifying selected soil sampling sites. Additional sampling sites, as selected by CDC, will be located at residences, parks, schools and playgrounds. Blood samples will be collected from pre-school aged children residing in each of the study sites.

Samples of the previously identified media will be collected according to the EPA and CDC sampling protocols provided as attachments 1 and 2. The samples will be prepared as prescribed in the sampling protocols. Chain-of-custody procedures as outlined in Section 5 of this plan, using the sample tags and chain-of-custody forms provided by the EPA On-Scene Coordinator will be followed. The samples will be transferred to the sample bank as soon as possible after collection.

Close communication will be maintained between the EPA QA Officer, sample bank personnel and field sampling team personnel. This on-site communication will be valuable in initiating and completing corrective action.

2.2 Sample Bank

The sample bank operation is being overseen by EPA personnel. Chain-of-custody documents will be dispensed from the sample bank, and custody records, from preparation of collected media through shipment of the collected sample, to analytical laboratories, will be maintained at the sample bank.

Media collecting equipment(containers, soil corers, dust collectors, etc.) will be dispensed to the sampling teams at the sample bank. QC samples, such as performance evaluation samples will be received at the saple bank from EPA or other preparing laboratories and inserted in the sample group to be shipped to the analytical contractors.

Collected samples will be accepted at the sample bank and logged into a master log. Since each sample will be assigned a new sample bank number and sample tag it will be possible to submit duplicate and spiked samples to analtical laboratories as blind QC samples, indistinguishable from colected samples. Sample bank operating procedures will be fully ducribed in attachment 1.

Sample bank personnel willarrange prompt shipment, usually by Federal Express, to the apropriate analytical laboratories. They will notify the laboratoryas to the expected date and delivery time of each shipment. Figure shows both the physical sample transfer into and out of the samplebank and the associated data flow.

2.3 Sample Analysis

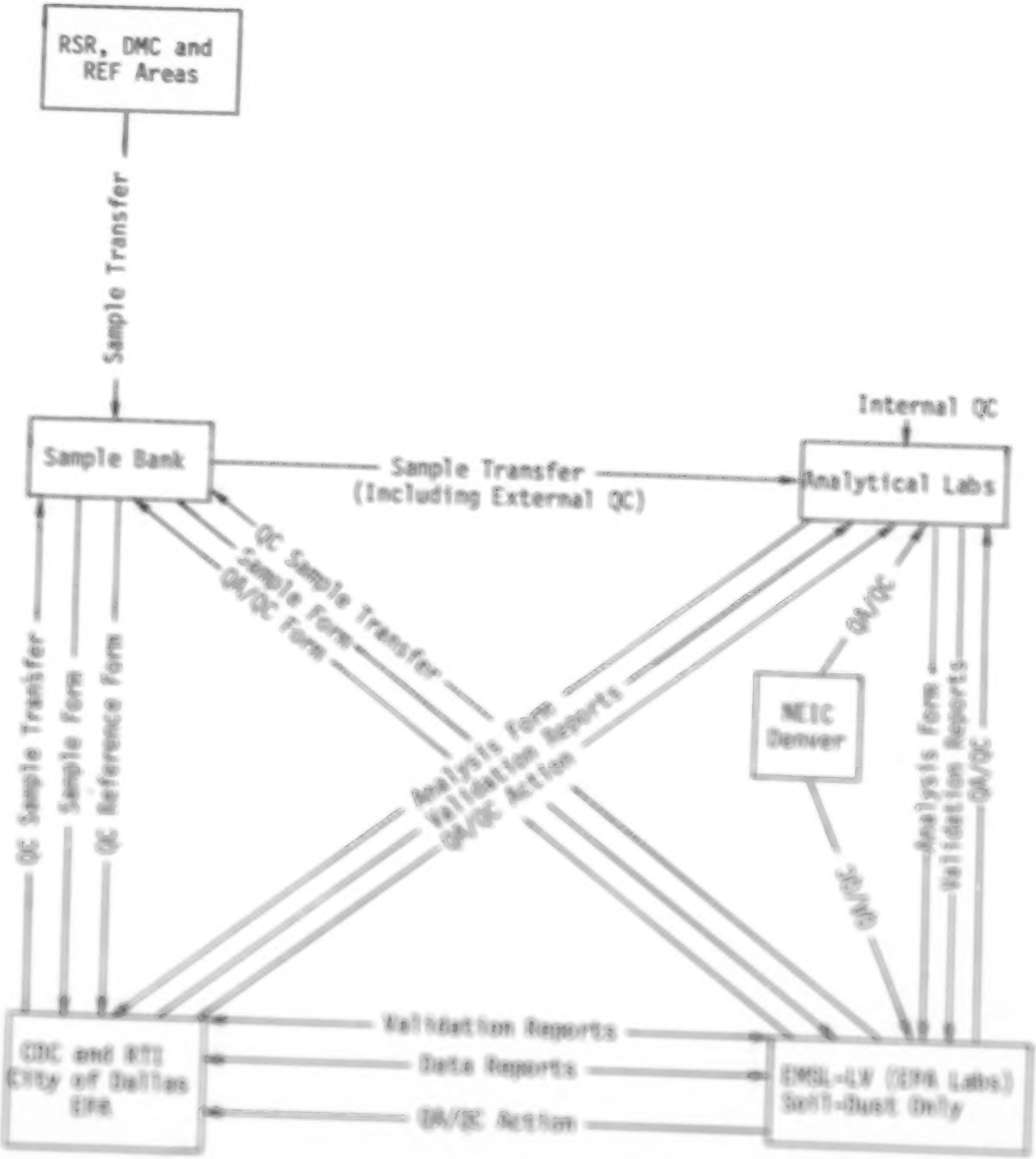

RSR, DMC and REF Areas
Sample Transfer
Sample Bank
Sample Transfer (Including External QC)
Internal QC
Analytical Labs
QC Sample Transfer
Sample Form
QC Reference Form
QC Sample Transfer
Sample Form
QA/QC Form
Analysis Form
Validation Reports
QA/QC Action
QA/QC
NEIC Denver
QA/QC
Analysis Form
Validation Reports
QA/QC
Validation Reports
Data Reports
QA/QC Action
CDC and RTI
City of Dallas
EPA
EMSL-LV (EPA Labs)
Soil-Dust Only

Figure 2. Sample Transfer and Data Flow.

The large number of analyses required for this program will be performed by the following laboratories:

City of Dallas	Dust
Contract	Soil

EPA will oversee the laboratories' work with special attention given to QC sample results, on-time completion of analyses and prompt submission of data. Close communication with the QA Project Coordinator at each laboratory will be maintained to monitor performance and determine if instrument downtime or other problems are preventing the anticipated throughput of samples. The sample bank will be notified promptly, throughout the program, of any changes in the sample shipping plan as required.

The analytical laboratories are responsible for notifying the sample bank of sample receipt and maintaining chain-of-custody records from receipt of the samples until completion of analytical work and transfer of all test data to EPA. In selected cases, sample extracts are to be preserved and returned with data.

2.4 QC Sample Analysis

EPA Laboratories will analyze replicate collected samples as noted below to establish interlaboratory precision.

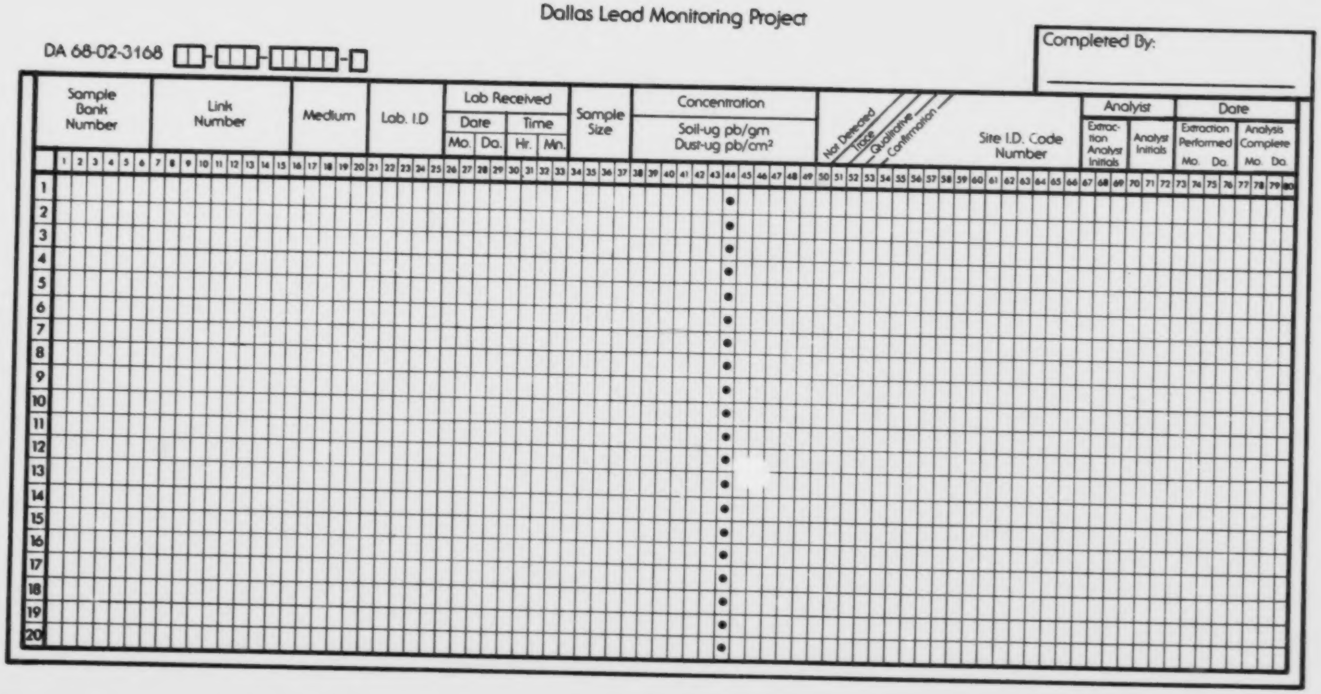
NEIC Denver	Soil-Dust
EMSL-LV	Soil-Dust (as specified)
[illegible]	

2.5 Data Handling

The sample bank will provide standardized sampling and analytical data reporting forms (Figure 3) which are designed in an 80-column format for direct keypunching of reported information. The reporting forms for sampling data will be coded at the sample bank so that all appropriate record numbers are entered. Each analytical laboratory will encode its own data and transmit the coding forms to CDC, RTI and EMSL-LV as shown in Figure 2.

Offices receiving data reports after validation based on their areas of interest.

EMSL-LV	Soil-Dust
CDC Atlanta	Paint-Soil-Dust
City of Dallas	Soil-Paint-Dust
EPA Region 6 Dallas	Soil-Paint-Dust
EPA Washington, D.C.	Soil-Paint-Dust



Dallas Lead Monitoring Project

DA 68-02-3168 ☐☐-☐☐☐-☐☐☐☐☐-☐

Completed By: ____________

Sample Bank Number	Link Number	Medium	Lab. I.D	Lab Received				Sample Size	Concentration	Not Detected	Trace	Qualitative	Confirmation	Site I.D. Code Number	Analyst		Date			
				Date		Time			Soil-ug pb/gm Dust-ug pb/cm²						Extraction Analyst Initials	Analyst Initials	Extraction Performed		Analysis Complete	
				Mo.	Da.	Hr.	Mn.										Mo.	Da.	Mo.	Da.

Rows 1–20; columns 1–80.

Figure 3. Sampling and Analytical Data Reporting Form.

3.0 QA OBJECTIVES

The QA objective of this monitoring program is to collect representative multimedia samples which can then be analyzed in a precise and accurate fashion with results reported in standard comparable formats.

3.1 Representative Sampling

EPA and CDC have designed the sampling protocols to collect a statistically valid number and geographical distribution of samples; the samplers are responsible for collecting samples representative of the media sampled. The sampling procedures, included as attachments 1 and 2 of this plan, provide the necessary guidelines for the conduct of the sampling program.

3.2 Precision and Accuracy

The QA objective is to achieve an intralaboratory precision (for the analysis only) of ±10% with a recovery of lead of 90 to 110%. The intralaboratory precision for the split samples is expected to be ±20%. For the overall precision and recovery (sampling, sample preparation and analysis) we expect ±20% and 80 to 120%, respectively. This may not be possible in all cases since the collected samples may contain varying interferences. The analytical laboratories have been provided with attachment 3 appropriate to their work and will report the precision and accuracy they achieve.

3.3 Comparability of Results

The measures taken to ensure comparability of analytical work performed by different laboratories include the use of:

- Standardized written sampling and analytical procedures.
- Standardized field and analytical data forms, and sample identification tags.
- All external QC samples of one type provided by one source.
- Standard handling and shipping procedures for all collected samples.

3.4 Dallas Lead Study Quality Assurance Plan

The overall QA Project Plan is contained in this plan and the following attachments:

Attachments 1 and 2:	Sampling Procedures
Attachment 3:	Analytical Procedures
Attachment 4:	RTI's Survey Design

This plan will be distributed to EPA CDC and the City of Dallas project personnel. The Sampling and Analytical attachments include the QC requirements specified by EPA and CDC and have been provided to the sampling and analytical laboratories as appropriate.

Each laboratory is required to submit to EPA a QA plan specific to his project activity. Appendix 1 to this plan presents the information submitted to the analytical laboratories as guidance in developing their QA plans. The Appendix includes format suggestions and suggested QA plan outlines.

Document control and chain-of-custody procedure writeups were also provided to each laboratory. Each laboratory's QA plan will be reviewed, revised if necessary and approved by EPA. The laboratories QA plans will become part of this QA plan as attachments.

4.0 DOCUMENT CONTROL PROCEDURES

4.1 General Information

The purpose of document control is to ensure that all project documents will be accounted for when the project is complete. Table 1 lists the principal items subject to document control with records maintained by the person listed as Document Control Officer (DCO) for that document. EPA will provide a DCO onsite at the sample bank who will maintain overall document control for all items passing through the sample bank.

Each laboratory shall identify a responsible person (DCO) for its operations who will be responsible for issuing, controlling and maintaining records of controlled documents. At the conclusion of laboratory activity, all controlled documents with records shall be provided to EPA for inclusion in an overall project document inventory.

4.2 Project Code

The project code for the Dallas Lead Study is DA 68-02-3168; this number must appear on sample and analysis identification tags,

TABLE 1. DOCUMENT CONTROL REQUIREMENTS

Item	Issued by	Numbering	Interim Responsibility	DCO Final Responsibility
Sample Collection Tags	Sample Bank	Preserialized	Sampling Team	Sample Bank
Custody Records	Sample Bank	Preserialized	See Section 5.0	See Section 5.0
Field Logbooks	City of Dallas		Sampling Team	Sample Bank
Site Description Forms	Sample Bank		Sampling Team	Sample Bank
Analytical Sample Tags	Sample Bank	Preserialized	Sample Bank	Analytical Laboratory
Laboratory Notebooks	Laboratory		Analytical Laboratory	Sample Bank
Analytical Data Sheets	Sample Bank		Analytical Laboratory	Sample Bank

custody records, logbooks, data sheets and other records and reports dealing with this project.

4.3 Preserialized Document

Sample collection and analytical tags, and custody records have preprinted serial numbers on them. It is not necessary that a tag number match a custody record number; it is necessary that each laboratory DCO maintain records which account for all serially numbered items received. If tags or custody records are damaged, lost or destroyed before use for their intended purpose, the serial number of the item and its disposition must be recorded.

5.0 CHAIN-OF-CUSTODY PROCEDURES

5.1 General Information

The purpose of chain-of-custody procedures is to document the identity of the sample, and its handling, from its first existence as a sample until information derived from it is introduced as evidence during an enforcement proceeding. Custody records trace a sample from its collection through all transfers of custody until it is transferred to an analytical laboratory. Internal laboratory records then document the custody of the sample through its final disposition.

In order to establish that a sample is valid, it is also necessary to document the measures taken to prevent and/or detect tampering--either to the sample itself, the sampling equipment used or the environment sampled. Documented or suspected tampering is to be described and recorded in the appropriate field and/or laboratory logbooks. The EPA Project Officer and On-Scene Coordinator should be notified immediately. Also, the custody record must reflect any tampering that may have occurred; the absence of any such comments indicates no tampering observed or noticed during the period of custody.

The sample bank will provide and dispense sample tags, custody records, logbooks, data sheets, etc. as appropriate. Custody procedures are presented here in considerable detail so that they may be uniformly followed.

The EPA shall identify an on-site person responsible for ensuring that custody procedures are followed and for referring any tampering cases or other questions to the On-Scene Coordinator. Analytical contractors shall also identify a person responsible for custody matters.

5.2 Sampling Equipment

The samplers must have documented proof that equipment has not been tampered with before, during and after sample collection. In many cases, this may be accomplished by placing strips of evidence tape on equipment storage boxes and storage rooms to demonstrate security of stored equipment. If tampering is suspected, document the situation including a judgment as to whether tampering was involved. Do not proceed with sampling until responsible personnel and the EPA on-site coordinator have been notified and EPA states that sampling should proceed for that day. Determine whether more stringent measures, such as locks or a guard, should be employed in the future at that site.

5.3 Sample Identification

Sample collection and analytical tags have been designed for soil and dust samples as shown in Figure 4. They are a different color for each media and are serially numbered starting with different blocks of numbers for each media and site (RSR, DMC and REF). The tags are preprinted to ensure that the required information is provided on each tag. Each collected sample, including duplicates and field blanks, shall have a completely filled-in sample tag securely attached to it. Duplicates and field blanks shall be so identified in the "Remarks" section of the tag along with other pertinent information.

The person who physically collects the sample is the Sampler and signs the sample tag. Another member of the team may enter the exact time of sample collection; all team members present shall be recorded in the logbook and on the site description form (see attachment 1). The Sampler initiates the custody record for transfer of samples to the sample bank.

After the collected samples have been transfered to the sample bank and documented there, they will be renumbered and identified with a sample analysis tag for shipment to analytical laboratories.

Project Code Dallas Lead	Soil	(Source of Sample) Grid Park School Home Playground Other	(Site Code)	
Address of Site (or Location)			Date	Time (Military)
Sampler (signature)				
Remarks: Sammple ID No. ____________			XXXX	

Sample Collection Tag

(Project Code) Dallas Lead	(Sample Bank No.)
(Sample Description)	
Analysis	
Metals (Lead)	
(Tag No.)	

Analysis Tag

Figure 4. Sample Collection and Analytical Tags.

This renumbering step enables the submittal of duplicate samples and QC samples as blinds to the analytical laboratory. The same analysis tag (Figure 4) is used for all samples; it is a different color and numbered with a block of numbers different from other tags.

The original sample collection tag number is recorded in the master log and the tag itself is stored in the document file at the sample bank. An important aspect of sample identification traceability is the system of cross-indexing which can correlate any one of the identifying numbers with a specific sample. These numbers include those found on the:

- Sample collection tag
- Custody record transferring custody to the sample bank
- Sample bank master log (sample bank number)
- Analytical sample tag
- Custody record transferring custody to the analytical laboratory.

5.4 Sample Transfer Procedures/Chain-of-Custody Records

There are many transfers of custody in this program since materials are shipped to the sample bank for distribution to the sampling team for inclusion with collected samples, and shipped from the sample bank to analytical laboratories. Laboratories providing QC samples must also initiate a custody record. Secure shipping procedures described in the Custody Seals Section should be followed for all shipments.

Chain-of-Custody Record Sheets

Customized record sheets, following the NEIC format, have been provided for this program and are shown in Figures 5, 6, and 7. The form shown in Figure 5 is used for samples collected in the field, the form shown in Figure 6 is for shipping QC samples to the sample bank and the form shown in Figure 7 is used for shipping samples to the analytical laboratories. They are two-part carbonless copy forms which correlate with the sample identification tags; requested information has the same heading on both.

The custody records are used for a packaged lot of samples; more than one sample will usually be recorded on one form. More than one custody record sheet may be used for one package, if necessary. Their purpose is to document the transfer of a group of samples traveling together; when the group of samples changes, a new custody

Environmental Protection Agency
Office of Enforcement

CHAIN OF CUSTODY RECORD

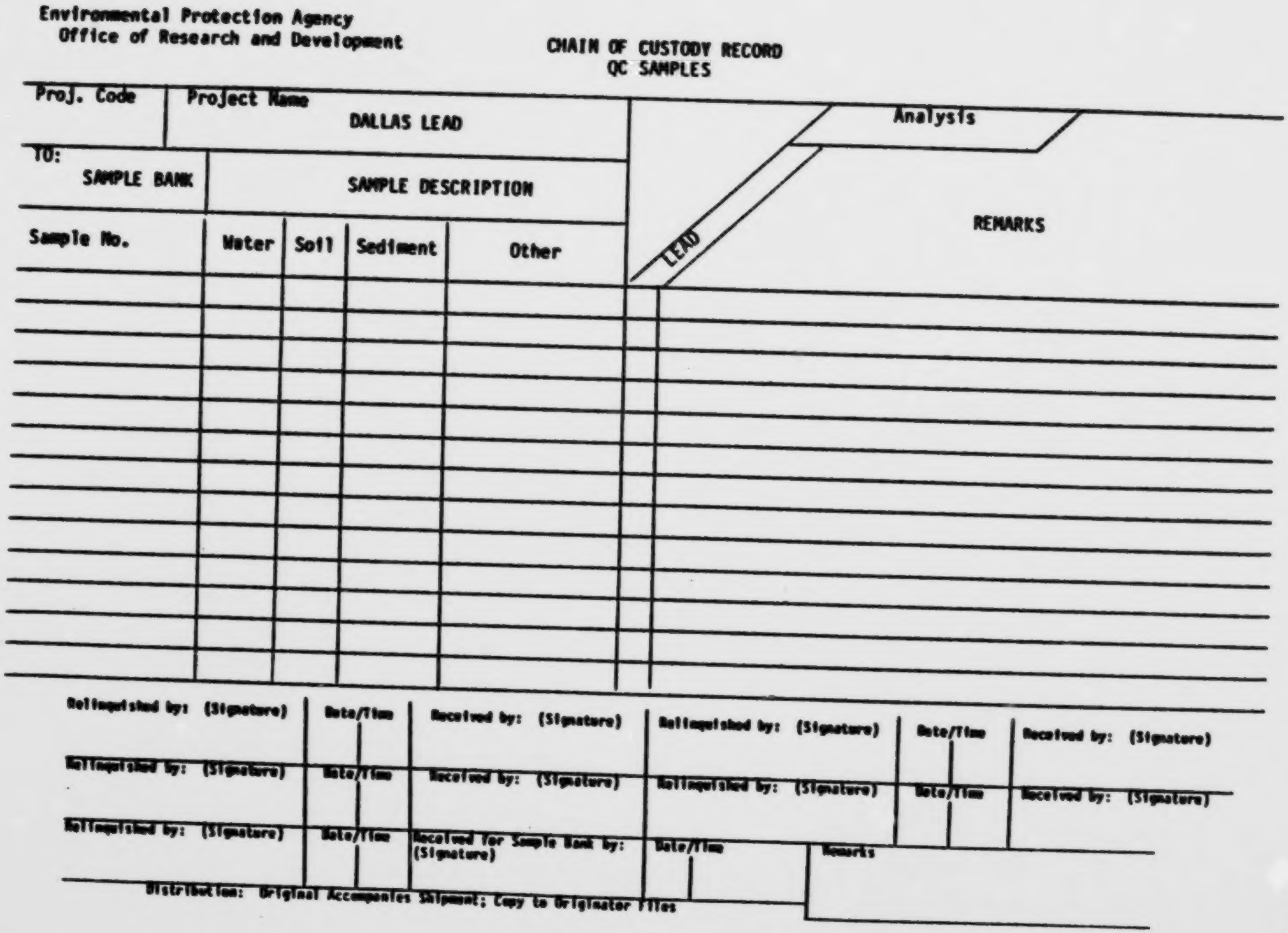
Proj. No.	Project Name DALLAS LEAD	No. of Containers	Analysis		Sample Type					REMARKS
SAMPLERS: (Signature)										

TAG NO.	DATE	TIME	COMP.	GRAB	SITE LOCATION	No. of Containers	LEAD		SOIL	DUST	BLOOD	VEGETATION	OTHER	REMARKS

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Sample Bank by: (Signature)	Date/Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Originator Files

Figure 5. Chain-of-Custody Record Sheet.



Environmental Protection Agency
Office of Research and Development

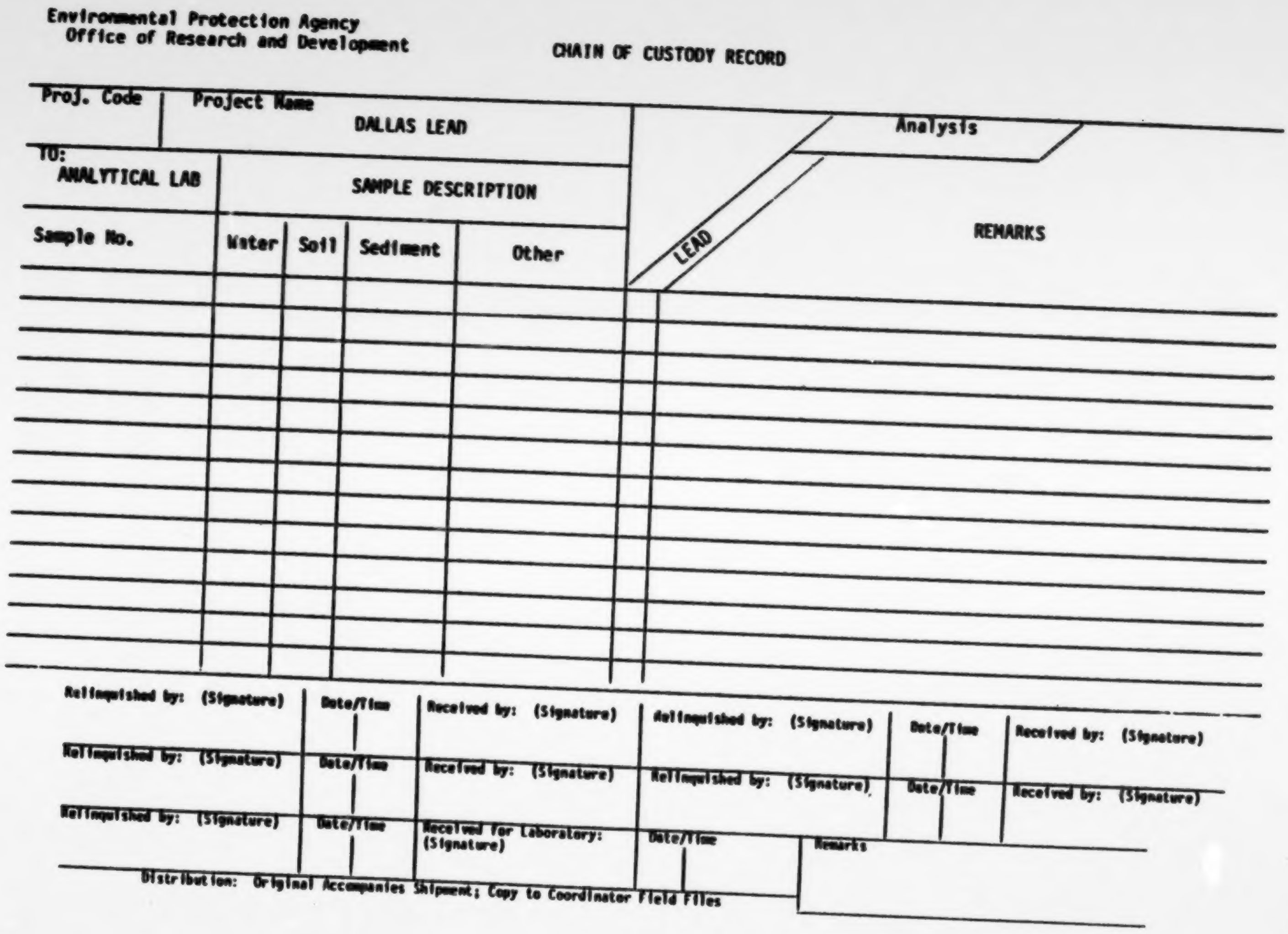
CHAIN OF CUSTODY RECORD
QC SAMPLES

Proj. Code	Project Name DALLAS LEAD				Analysis	
TO: SAMPLE BANK	SAMPLE DESCRIPTION					
Sample No.	Water	Soil	Sediment	Other	LEAD	REMARKS

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Sample Bank by: (Signature)	Date/Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Originator Files

Figure 6. Chain-of-Custody Record Sheet.



Environmental Protection Agency
Office of Research and Development

CHAIN OF CUSTODY RECORD

Proj. Code	Project Name DALLAS LEAD	Analysis

TO:
ANALYTICAL LAB

Sample No.	SAMPLE DESCRIPTION Water	 Soil	 Sediment	 Other	LEAD	REMARKS

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Laboratory: (Signature)	Date/Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

Figure 7. Chain-of-Custody Record Sheet.

record is initiated. The original of the custody record always travels with the samples; the initiator of the record keeps the copy. When custody of the same group of samples changes hands several times, some people will not have a copy of the custody record. This is acceptable as long as the original custody record shows that each person who had received custody has properly relinquished it.

Using the Custody Record Sheet

- The originator fills in all requested information from the sample tags.

- The person receiving custody checks the sample tag information against the custody record. He also checks sample condition and notes anything unusual under "Remarks" on the custody form.

- The originator signs in the top left "Relinquished by" box and keeps the copy.

- The person receiving custody signs in the adjacent "Received by" box and keeps the original.

- The Date/Time will be the same for both signatures since custody must be transferred to another person.

- When custody is transferred to the sample bank or an analytical laboratory, blank signature spaces may be left and the customized last "Received by" signature box used. Another approach is to run a line through the unused signature boxes.

- In all cases, it must be readily seen that the same person receiving custody has relinquished it to the next custodian.

- If samples are left unattended or a person refuses to sign, this must be documented and explained on the custody record.

Analytical Samples

- Initiated by sample bank.

- Custody relinquished by shipper, sample bank, before packing the original of custody form in with the samples.
- Sample bank keeps copy of custody form and all shipping papers, bills of lading, etc.
- Custody transferred to analytical lab which keeps original record.
- Analytical Lab Document Control Officer maintains original custody record until transmittal of complete project document inventory to EPA at completion of project activity.

Questions/Problems Concerning Custody Records

If a discrepancy between sample tag numbers and custody record listings is found, the person receiving custody should document this and properly store the samples. The samples should not be analyzed until the problem is resolved by contacting the EPA On-Scene Coordinator or other designated responsible authority.

shipper (e.g., sample bank personnel) accompanies the package to the carrier (e.g., Federal Express) so that, if requested, the number and identification of the samples in the container can be verified. The commericial carrier is not required to verify this.

The package is then closed with a strong, keyed padlock or strapping tape and custody seals so that the carrier is transporting a secure container.

NEIC's recommended procedure for custody seal use on shipping boxes is as follows:

- Place paper side of seal against box and wrap strapping tape across the middle of the seal and at least twice around the box.
- Cut the tape so that the ends overlap at the seal.
- Fold the seal to itself over this overlap and sign the custody seal.
- Repeat this procedure as many times as necessary to seal the box for shipment.

The responsible person receiving custody should attempt to resolve the problem by checking all available information (other markings on sample container, type of sample, etc.). He should then document the situation on the custody record and in his project logbook and notify the EPA On-Scene Coordinator by the fastest available means, followed by written notification.

Changes may be written in the "Remarks" section of the custody record and should be initialed and dated. A copy of this record should accompany the written notification to the EPA On-Scene Coordinator.

Custody Seals

Custody seals are narrow strips of adhesive paper used to demonstrate that no tampering has occurred. They may be used on sampling equipment but are intended for use on a sample transport container which is not secured by a padlock. They are not intended for use on individual sample containers.

Custody of Shipped Samples

When a group of samples with its custody form is to be shipped, the

The person receiving custody of shipped samples must document the condition of the locked, or strapped and sealed shipping box on arrival. It must be checked that neither the tape or the custody seals have been cut or otherwise tampered with. If the paper seal has been damaged in shipping but it is clear that the shipping box has not been opened, further handling of the samples may proceed. If tampering is suspected, the designated Sample Custodian shall notify the On-Scene Coordinator.

5.5 Laboratory Custody Procedures

The laboratory shall designate a "sample custodian." An alternate will be designated in his absence. In addition, the laboratory shall set aside a "sample storage security area." This should be a clean, dry, isolated room which can be securely locked from the outside.

All samples should be handled by the minimum possible number of persons.

All incoming samples shall be received only by the custodian, who will indicate receipt by signing the Chain-of-Custody Record Sheet accompanying the samples and retaining the sheet as permanent records. Couriers picking up samples at the airport, post office, etc. shall sign jointly with the laboratory custodian.

Immediately upon receipt, the custodian will place the sample in the sample room, which will be locked at all times except when samples are removed or replaced by the custodian. To the maximum extent possible, only the custodian should be permitted in the sample room.

Only the custodian will distribute samples to personnel who are to perform tests.

Laboratory personnel are responsible for the care and custody of the sample once it is handed over to them and should be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the tests were run.

Once the sample testing is completed, the unused portion of the sample together will all identifying tags and laboratory records, should be returned to the custodian. The returned tagged sample will be retained in the sample room until it is required for trial. Strip charts and other documentation of work will also be turned over to the custodian.

6.0 SAMPLING INTERNAL QC CHECKS

6.1 Soil and Dust

The sampling program includes the collection of soil and dust samples. Attachments 1 and 2 present the sampling procedures to be followed for collecting each type of sample and incorporate information on appropriate sample collection equipment, types of sample containers and their cleanup, preservation required, field blanks and chain-of-custody procedures.

Table 2 shows the summarized internal QC measures to be used in the soil and dust sample collection program.

TABLE 2. SOIL AND DUST SAMPLING INTERNAL QC CHECKS

Media	Pollutants Measured	Operation Check	Control Limits	Planned Corrective Action
Soil	Lead	1. Prepare under field conditions one DDI water field blank for every 20 soil samples collected.	1. No significant concentrations found.	1a. Correct for field blank. 1b. Check sampling and analytical procedures. 1c. Invalidate results.
		2. Collect 5% of samples in duplicate.	2. None	2. None
		3. Collect 5% of samples for splits.	3. None	3. None
Dust	Lead	1. Prepare under field conditions one cotton swab for every 20 dust samples collected.	1. No significant concentrations found.	1. Correct for field blank.

7.0 ANALYTICAL INTERNAL QC CHECKS

7.1 General Information

The analytical program includes the analysis of soil and dust samples that have been transferred to the laboratory under chain-of-custody. Attachment 3 presents the analytical procedures and internal analytical quality control procedures to be followed by the analytical laboratories.

7.2 Soil and Dust

Table 3 shows the summarized internal QC measures to be used in the soil and dust analytical program. The frequency of asterisked Operation Checks are subject to revision after the initial 200 to 400 samples are analyzed.

TABLE 3. SOIL AND DUST ANALYTICAL INTERNAL QC CHECKS

Media	Pollutants Measured	Operation Check	Control Limits	Planned Corrective Action
Soil	Lead	1. Calibration and standardization- Calibrate and standardize instrument as required in analytical protocol. Analyze a Calibration Control Sample (CCS) immediately after calibration and after analysis of every 20 soil samples.	1. Analytical Protocol and Standard Analytical Methods. Calculate CCS as percent true value. Maintain control chart for CCS and develop the 99 and 95% confidence limits, (control and warning). Acceptable range is the lesser of 95 to 105% at the 99% confidence limits.	1a. Recalibrate and restandardize instrument (including fresh stock and working standard, if necessary). 1b. Analyze a second Calibration Control Sample 1c. Invalidate results and repeat analyses.
		2. Reagent Blank - Analyze 3 per shift day.	2. No significant concentrations found ($\leq$0.25 µg/ml).	2a. Check for reagents and glassware contamination 2b. Correct for blank 2c. Contact EMSL-LV
		3.* Spiked Extract - to one (1) of every twenty (20) sample extracts, add standard level nitrate solution. Spike level is to be approx. equal to the endogenous level or ten (10) times the instrument detection limit.	3. Recovery for lead is 90-110%.	3a. Repeat on two additional sample extracts. 3b. If either or both of reagents are out side control limits, analyze 20 extracts by method of standard additions.[a]

(continued)

TABLE 3. (Continued)

Media	Pollutants Measured	Operation Check	Control Limits	Planned Corrective Action
Soil	Lead	4.* Residue Reextraction - For every twenty (20) soil samples extracted, reextract and analyze one (1) sample residue.	4. Calculate % extraction (first extraction) limits to be determined. The initial limit is 90%. Limit will be relieved, if necessary, after analysis of the first 200 to 400 samples.	4a. Check analytical procedure. 4b. Analyze laboratory control standard. 4c. Invalidate results.
		5.* 'Total Recoverable' Analysis - For every twenty (20) soil samples, one sample is extracted and analyzed using the 'Total Recoverable' analysis method in the analytical protocol.	5. Compare 'Total Recoverable' results to extraction method results. Resuls to agree within 10%. Limits will be relieved, if necessary, after analysis of the first 200 to 400 samples.	5a. Check analytical procedure. 5b. Analyze a second sample by 'Total' method. 5c. Invalidate results.

(continued)

TABLE 3. (Continued)

Media	Pollutants Measured	Operation Check	Control Limits	Planned Corrective Action
Soil	Lead	6.* Laboratory Control Sample (LCS) - Analyze one (1) LCS for every twenty (20) soil samples. If the first 10 results meet the control limits, reduce to one per shift. LCS to consist of 5 grams of NBS 1645 sediment.	6. Calculate - LCS as percent true value. Maintain control chart for LCS and develop the 99 and 95% confidence limits (control and warning). The acceptable recovery range is 90-110% of the 99% confidence limits, whichever is less.	6a. Check Analytical Procedure. 6b. Analyze second LCS sample. 6c. Invalidate results.
		7.* Split Extract Analysis - Analyze a second aliquot of one (1) of every twenty (20) soil sample extracts.	7. A precision of 3% is expected. If the precision is outside of the 5% range, EMSL-LV must be notified immediately.	7a. Recalibrate as in 1. 7b. Analyze a second sample extract in duplicate.
		8.* Spiked Sample Analysis - For every twenty (20) field samples, a duplicate portion of one sample is spiked with lead at a level approx. equal to the endogenous level but at least 10 x detection limit.	8. Calculate % recovery. Maintain control charts on the percent recovery and develop the 99 and 95% confidence limits (contol and warning). Acceptable range for lead recovery is 90 to 110% a the 99% confidence limits. Acceptable range to be reviewed after initial 200 samples.	8a. Check analytical procedure. 8b. Repeat spiked sample analysis on an additional sample. 8c. Contact EMSL-LV for assistance.

(continued)

TABLE 3. (Continued)

Media	Pollutants Measured	Operation Check	Control Limits	Planned Corrective Action
Dust	Lead	1. Calibration and Standardization - Calibrate and standardize instrument as required in analytical protocol. Analyze a Calibration Control Sample (CCS) immediately after calibration and after analysis of every twenty (20) samples.	1. Analytical Protocol and Standard Analytical Methods[a], calculate CCS as percent true value. Maintain control chart for CCS and develop the 99 and 95% confidence limits (control and warning). Acceptable range is the lesser of 95 to 105% at the 95% confidence limits.	1a. Recalibrate and restandardize instrument (including fresh stock and working standard, if necessary). 1b. Analyze a second Calibration Control Sample 1c. Invalidate results and repeat analyses.
		2. Reagent Blank - Analyze three (3) per shift day.	2. No significant concentrations found ($\leq$ 0.25 μg/ml).	2a. Check for reagent and glassware contamination. 2b. Correct for blank. 2c. Contact EMSL-LV.

(continued)

TABLE 3. (Continued)

Media	Pollutants Measured	Operation Check	Control Limits	Planned Corrective Action
Dust	Lead	3.* Spiked Extract - to one (1) of every twenty (20) sample extracts, add standard level nitrate solution. Spike level is to be approx. equal to the endogenous level or ten (10) times the instrument detection limit.	3. Recovery for lead is 90-110%.	3a. Repeat on two additional sample extracts. 3b. If either or both of reagents are outside control limits, analyze 20 extracts by method of standard additions.[a]
		4.* Laboratory Control Sample (LCS) - Analyze one (1) LCS for every twenty (20) dust wipe samples. If the first 10 results meet control limits, reduce to one per shift. LCS to consist of 0.1 gram NBS 1648, Urban Particulate and two freshly unwrapped dust wipe swabs.	4. Calculate LCS as percent true value. Maintain control chart for LCS. Develop 99 and 95 percent confidence levels (control and warning). Acceptable recovery range is 90 to 110% at the 99% confidence limits, whichever is less.	4a. Check analytical procedure. 4b. Analyze second LCS sample. 4c. Invalidate results.

(continued)

TABLE 3. (Continued)

Media	Pollutants Measured	Operation Check	Control Limits	Planned Corrective Action
Dust	Lead	5.* Split Extract Analysis - Analyze a second aliquot of one (1) of every twenty (20) dust sample extracts.	5. A precision of 3% is expected. If the precision is outside of the 5% range, EMSL-LV must be notified immediately.	5a. Recalibrate as in 1. 5b. Analyze a second sample extract in duplicate.

[a] Methods for Chemical Analysis of Water and Wastes, EPA-600-4-79-020. Atomic Absorption Spectroscopy procedures for metals.

*The frequency of these analyses may be decreased during the study.

8.0 ACCURACY

8.1 General Information

This section describes the accuracy of analysis which is a function of precision and bias and the measures planned by EPA and CDC for this study to determine these factors.

Although there are apparent differences between the numbers of replicate samples collected for precision estimates, the plan as stated is appropriate to the media sampled. A higher percentage of field replicate soil samples is collected than are collected for dust because dust samples collected on cotton swabs cannot be split under field conditions.

The following sections describe the precision and bias determinations planned for each media (soil and dust) sampled and analyzed.

8.2 Soils

Precision

Precision for soil sampling will be estimated from the analysis results of triplicate (split) samples collected at 5 percent of the sampling locations, and actual duplicates collected at an additional 5 percent of the sampling locations.

Intralaboratory precision for each analytical laboratory will be estimated from its analysis of duplicates from these triplicate (split) samples as well as from actual duplicate sample analysis.

Interlaboratory measurement precision will be determined from analysis of the third replicate in each triplicate set by the referee laboratory, NEIC Denver.

Intralaboratory precision for analysis only will be estimated from the results of carrying two replicate aliquots (split extract) from every tenth sample through the entire analytical procedure.

Bias

Analytical bias for soil analyses will be estimated from each contracting laboratory's results on Laboratory Control Samples (LCS). These quality control samples will be provided by EMSL-LV. After the initial calibration, one LCS will be analyzed for each 20 collected samples received.

8.3 Dust

Precision

Overall measurement precision cannot readily be estimated for dust samples since duplicates cannot be provided. However, the analytical laboratories will split the extract for approximately 5 percent of the dust samples into 3 parts. One part of the prepared extract will be sent to the referee laboratory, NEIC Denver, the other two parts will be analyzed by the contractor.

Intralaboratory precision for analysis only will be estimated from the results of carrying two replicate aliquots (split extracts) from every twentieth sample through the entire analytical procedure.

Bias

Analytical bias for dust sampling will be estimated from each laboratory's results on Laboratory Control Samples. These control samples will be provided by EMSL-LV. One LCS sample will be analyzed for each batch of approximately 20 collected samples. Laboratory internal QC data on Laboratory Control Standards will be used to calculate expected bias.

8.4 Accuracy Equations

The precision of analytical results on duplicate or triplicate collected samples shows the variability present in the sampling, sample preparation and measurement system; the analytical results on known value QC samples shows the bias present in the analytical measurement. The equations used to estimate precision and bias are given in the following sections.

Intralaboratory Precision Equations

Intralaboratory measurement precision for each analytical laboratory will be estimated from its analysis of duplicate collected samples. For each duplicate sample we assume that the following mathematical model holds:

Y_i and X_i are independent, normally distributed variables both with mean μ_i and standard deviation σ. We assume the mean μ_i can change from day to day but σ is constant.

The intralaboratory precision is quantified by σ and it is estimated by computing the mean of the differences and dividing by 1.128 (the factor for converting range to standard deviation for two samples). The following equations are used for either duplicate samples or duplicate extracts:

Difference Between Duplicates

$$\% \, d_i = \frac{2\,(Y_i - X_i)}{Y_i + X_i} \times 100 \qquad (1)$$

where Y_i = concentration measured by duplicate 1,
X_i = concentration measured by duplicate 2 and
d_i = difference between duplicate analyses.

$$\overline{D} = \frac{1}{k} \sum_{i=1}^{k} d_i \qquad (2)$$

where $\overline{D}$ = mean % difference
k = number of duplicates

Standard Deviation of Differences (S_d)

$$S_d = \overline{D}/1.128 \qquad (3)$$

Intralaboratory Precision Reporting

For each set of split extracts analyzed, the following will be reported:

y values, x values, differences

$\overline{D}$, mean difference

Upper and Lower 95% Probability Limits ($\pm 1.96\ S_d$)

Interlaboratory Precision Equations

Intralaboratory measurement precision for each laboratory will be estimated from the analysis of duplicate or triplicate splits from collected samples by the analytical laboratory and the EPA referee laboratory. As noted, soil split samples will be provided in triplicate while dust sample split extracts will be prepared for analysis to provide triplicates for precision estimates. The laboratory precision is quantified by the standard deviation of the difference between the subcontractor analysis results and the referee laboratory results.

Triplicate Samples

Two of the triplicates will be analyzed by the contractor and the other by the EPA referee laboratory; the variability between these measurements is the interlaboratory precision. The mathematical model appropriate here is that the differences, $d_i = Y_i - X_i$ are independent, normally distributed variables with mean μ_d and standard deviation σ_d. D is an estimate of the mean μ_d.

Ideally $\mu_d = 0$. If this is not true then there is a bias (on the average the analytical contractor's values do not equal the referee laboratory's values) and $\overline{D}$ is a point estimate of this bias. The standard deviation σ_d is the parameter used to quantify the interlaboratory precision and is estimated by the sample standard deviation S_d which is calculated using the following equations:

Differences Between Subcontractor and Referee Laboratory Results

$$\% \, d_i = \frac{2\,(Y_i - X_i)}{Y_i + X_i} \times 100 \qquad (4)$$

where Y_i = subcontractor analysis result (see note),

X_i = referee laboratory analysis result and

d_i = difference between duplicate analyses.

Note: There will be 2 d_i's for each sample as the contractor will be analyzing two extracts, the referee laboratory analyzes one.

$$\overline{D} = \frac{1}{k} \sum_{i=1}^{k} d_i \tag{5}$$

where $\overline{D}$ = mean difference

k = number of duplicates

Standard Deviation of Differences (S_d)

$$S_d = \overline{D}/1.128 \tag{6}$$

Assuming a normal distribution, 95 percent of the differences, d, are estimated to fall between

$$\overline{D} + 1.96\ S_d \text{ and } \overline{D} - 1.96\ S_d \tag{7}$$

These values can be used as control limits on the d's if so desired.

Interlaboratory Precision Reporting

For each set of duplicates analyzed, the following will be reported by the EMSL-LV or the contractor as appropriate:

y values, x values, differences

$\overline{D}$ mean difference = point estimate of bias

S_d, standard deviation of differences

± 1.96 S_d, for Upper and Lower Control Limits

Bias Equations

Analytical bias for each contractor laboratory will be estimated from its analysis results on external QC samples whose true value is known. The mathematical model appropriate here is that values of d_j are independent and normally distributed with mean μ_b and standard deviation σ_b. If the contractor has no bias then

$$b_j = Y_j - X_j = 0 \text{ identically}$$

and

$$\mu_b = 0 \text{ identically.}$$

If $b_j \neq 0$ and if $\mu_b \neq 0$ the contractor's results are not completely accurate and a bias exists. We define μ_b as the bias; its point estimate is $\overline{B}$. The following equations are used to calculate $\overline{B}$.

Difference from True Value, or Bias

$$b_j = Y_j - X_j \qquad (8)$$

where Y_j = contractor analysis result,

X_j = true value, and

b_j = difference from true value.

$$\overline{B} = \frac{1}{n} \sum_{j=1}^{n} b_j \qquad (9)$$

where $\overline{B}$ = mean difference = point estimate of bias

n = number of external QC samples analyzed

95 Percent Confidence Interval

The point estimate $\overline{B}$, can be expanded to an interval estimate or confidence interval:

$$\overline{B} \pm t \frac{S_b}{\sqrt{n}}$$

where

$$S_b = \sqrt{\frac{1}{(n-1)} \left[\sum_{j=1}^{n} b_j{}^2 - \frac{1}{n} \left(\sum_{j=1}^{n} b_j \right)^2 \right]} \qquad (10)$$

and t is the appropriate value to produce the confidence interval with desired confidence level. For example, if $n \geq 30$ and a 0.95 confidence interval level is desired, then $t = 1.96$. If the confidence interval contains zero, we can say with 95 percent confidence that the difference is caused by random variation and not by a bias in the measurement.

Bias Reporting

For each external QC sample analyzed, the following will be reported:

y values, x values, differences
$\overline{B}$, mean difference = point estimate of bias
S_b, standard deviation of differences
$\overline{D} \pm t \frac{S_b}{\sqrt{n}}$, Upper and Lower 95 percent Confidence Limits

Recovery

Recovery is defined as the amount recovered divided by the amount added (or true value). This can be converted to percent recovery by multiplying by 100.

$$\% \text{ Recovery} = \frac{\text{Amount Recovered}}{\text{True Value}} \times 100 \qquad (11)$$

Range of a Reported Value

The concentration of an analyte in a sample as reported by the analytical laboratory is an estimate because of the bias and precision of the method used. The true value in that sample will be unknown, but upper and lower limits for the concentration can be calculated as follows, using a 95% confidence interval:

Upper 95% Limit = X_i (1-B - 2 S_d)

Lower 95% Limit = X_i (1-B - 2 S_d)

Where X_i = reported concentration,

B = $\overline{B}$ from Equation (9)

S_d = S_d from Equation (3) for single laboratory result,

= S_d from Equation (6) for result independent of laboratory.

9.0 DATA MANAGEMENT SYSTEM

9.1 General Information

The purpose of the data management system is to collect and store all data generated as part of the Dallas Lead Study and to provide basic reports of the monitoring results.

Development of a system to manage and report the sampling data will require activity in five general areas:

- Defining data flow and traceability
- Preparation of data coding forms
- Creation of a Manual of Codes and Data Coding Procedures
- Development of handling and reporting software
- Generation of tapes of raw data, verified data, and validated data for use by EPA and CDC.

9.2 Data Description

Data Flow

Figure 8 shows the flow of data through the system. The maintenance of three separate data files for raw, verified and validated data should be noted.

The difference among these files is the level of review involved in ensuring accuracy of the data. The raw data file represents data which have received a cursory review to confirm that the data have been provided in the proper format. At the raw data level, all key data are correct and all mandatory data supplied.

At the verified data level, the information has all been reviewed by the supplying group (e.g., the analytical laboratories) and any errors in transcription, keying, or processing corrected.

The validated data file corresponds to a much higher level of review. All QC runs, both internal and external, are reviewed in relation to the analytical concentrations and corrections to the data made. The data in this file will be supplied to CDC and EPA for further review.

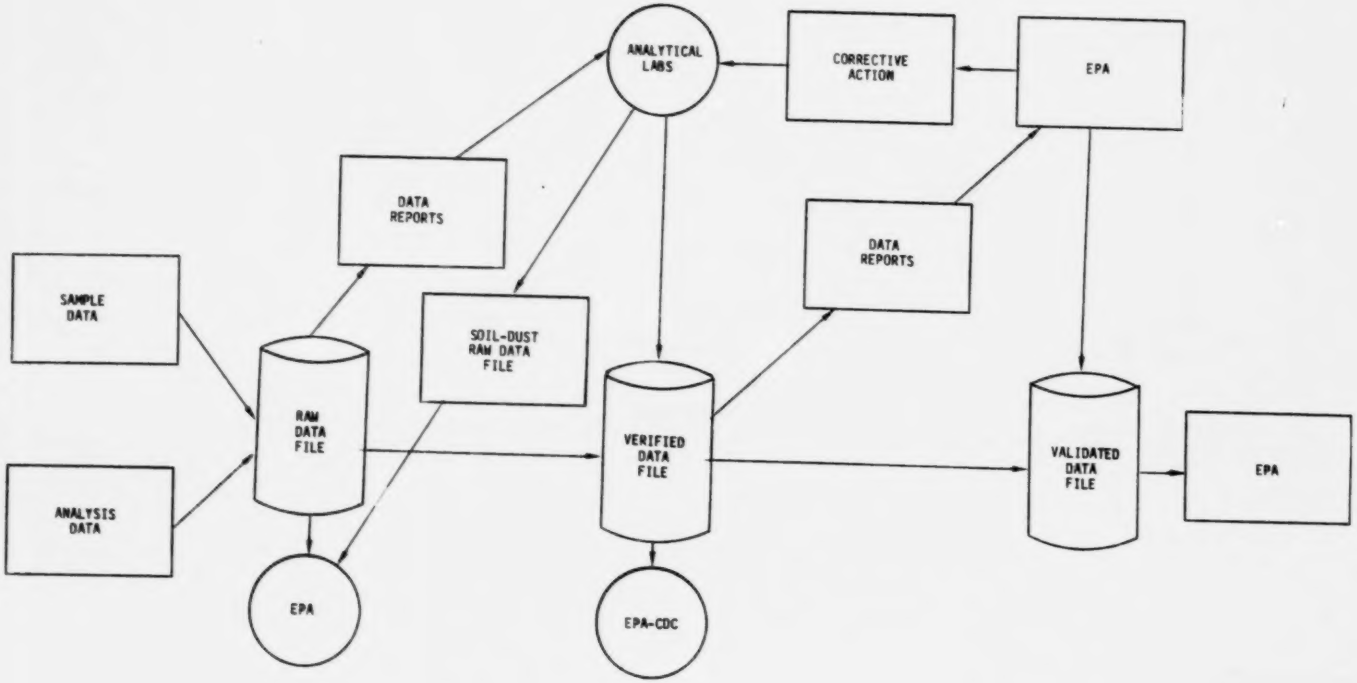

ANALYTICAL LABS
CORRECTIVE ACTION
EPA
DATA REPORTS
SAMPLE DATA
DATA REPORTS
SOIL-DUST RAW DATA FILE
RAW DATA FILE
VERIFIED DATA FILE
VALIDATED DATA FILE
EPA
ANALYSIS DATA
EPA
EPA-CDC

Figure 8. Data flow diagram.

9.3 Input Data

Input of all data to the system will be done by use of computer cards. The data to be stored in the system have been categorized into two areas:

- Sample data
- Analytical data

In addition, the following special information is required:

- Duplicate (QA/QC) cross-reference data
- Verification data
- Validation data

1. Sample data - Sample data include information such as sample type, location, time, date, and tag numbers. The sample data coding form will originate in the sample bank, using data supplied by the field collection teams. The master sample

number will be added to the form along with the analytical lab to which the sample is being sent and other appropriate custody or tag numbers.

This sample data recorded on the form, will be keypunched and entered into the data handling system. It will then serve as the basis for generating a tag number/custody number/master ID cross-reference system which will be provided to the sample bank on a weekly basis. This form will also serve as a basis for generating deliquency reports for analytical samples which are overdue.

Analytical Data

A form submitted to the analytical laboratory by the sample bank will be completed for each sample by the analytical lab. The labs will also complete coding forms for QC samples and procedures which they provide. These samples will be entered into the data system after being assigned a sample number.

These forms will be sent to the appropriate EPA lab, to RTI and to CDC. At RTI, they will be keypunched.

Duplicate Cross-reference Data

The sample bank will submit duplicate or triplicate samples to analytical laboratories as part of the QA/QC program. Each time this is done, a special form giving the replicate sample ID numbers, plus any comments as to why the sample was chosen, will be coded and sent to RTI for keypunch. The replicate samples will then be reported adjacent to the first samples. The first sample of the replicate will have the lowest sample bank number.

Verification Procedures

RTI will provide a raw data listing to each analytical lab and other responsible labs, listing sample information together with analysis data for samples analyzed by that lab. Possible data problems such as missing values, high concentrations, low check sample recoveries, etc., will be highlighted by RTI on this computer listing. The labs will specify corrective action to be applied to the raw data file and RTI will then code forms to accomplish these corrections. It is expected that the principal actions will be "ADD," "DELETE," or "REPLACE" and the form will be set up to handle these functions. More complex directions, such as "double the value," will require special programmer actions. This form will serve as a record of who made changes, and it will include any notation as to why the changes were necessary.

Validation Procedures

Final instructions for creation of the validated file are expected to be relatively complex and the form of the instructions, other than a "delete" function, will most likely have to be applied on a case-by-case basis. Hence, no specific form is envisioned for this purpose. Information will be collected from responsible parties and appropriate software developed and applied to create the validated data file.

Output Data

A number of reports will be generated from the raw data, the verified data, and the validated data files. Raw data files, verified data files, and validated data will be transferred to EMSL-LV and CDC for their use. The reports to be developed are described below.

ID Number Cross-reference

Each sample may have from four to eight different tag/custody numbers associated with it. The sample bank sample number will serve as the basis for cataloging all of these numbers. The ID Number Cross-reference report will provide all necessary cross-referencing to determine the master number if some other number (e.g., sample tag number) is known. It will be updated weekly and stored at RTI and the sample bank.

Data Summaries

A listing of all data received during the last processing period (expected to be 1-week intervals) will be generated and sent to the appropriate EPA, CDC and analytical labs for review. Each file will have a header describing the data file accessed plus other pertinent information.

These summaries will be generated in three ways:

- By sample
- By laboratory and sample
- By sample and location (verified data only).

Other summaries can be developed if requested.

Alert Reports

An alert level will be determined. A report of all analytes by sample which exceed this value will be generated. This report will provide a focus for special attention to identify bad data or actual high concentration areas.

Delinquency Report

For each lab, the RTI computer will generate a list of all samples for which it has not provided analysis data within the prescribed time period. This report will serve as the basis for a letter to the lab, reminding it of these requirements.

Intermediate and Final Data Transfer

The raw data, verified data, and validated data files will be transferred to EPA and RTI in readable formats.

9.4 Data Management Internal QC Checks

The key components of the data management QC program include the following items:

- Development of uniform data reporting forms
- Issuance of data reporting/codes manuals to all parties
- Keypunch verification of all input data
- Online editing of all input data fields with error reports being issued for review.
- Log-in of all data report forms received at RTI.

10.0 SYSTEM AND PERFORMANCE AUDITS

10.1 General Information

Throughout the program, audits will be conducted by EPA personnel and by CDC personnel. In addition, RTI will audit its own data management system and anticipate unannounced audits by EPA or a third party.

10.2 System Audits

Sample Collection Audits

The EPA QA Manager will audit the overall QA system for sample collection, preparation storage, and transfer just before sampling starts. The intent of this audit is to critically review the entire onsite operation to determine the need for any corrective action early in the program. Additional audits will be conducted during the sampling program.

EPA will maintain a QA Coordinator onsite during sample collection to monitor the sampling team's activities, provide technical assistance and corrective action suggestions to the sampling teams, and implement performance audits on sampling as needed. The frequency of system audits by the QA Manager will be determined, in part, from input from EPA's S&A Division Chief in Region 6, from the City of Dallas QA Coordinator and from other regional field personnel.

Chain-of-Custody Audits

EPA personnel will conduct a system audit of the custody system in use on this project. EPA QA personnel will trace specific samples through the system from time to time.

Analytical Audits

Site visits to contractor analytical labs will be made by EPA and EPA designated personnel on a biweekly basis during active analytical work. Frequent communication between EPA analytical QA coordinators and each analytical contractor will be maintained throughout the program.

Data Management Audits

EPA QA Coordinator will audit the data management system by tracing the flow of specific samples through the system. In particular, the ability of the system to correctly identify a sample from any one of its identification numbers will be checked. Copies of the sample bank's logbook pages will be the basis for these performance checks. From time to time, erroneous input information may be used to audit the system.

10.3 Performance Audits

Sample Collection Audits

EPA personnel will audit field blanks collected for soil and dust samples throughout the program.

Analytical Audits

Section 8.0 of this volume describes the planned accuracy checks on the analytical contractor's work.

11.0 CORRECTIVE ACTION PROCEDURES

11.1 Determining the Need for Corrective Action

The need for corrective action may be identified by standard QC procedures, control charts, performance or system audits. Often the experience of a field technician or an analyst will be most valuable in alerting him to malfunctioning equipment or suspicious data.

To promptly and uniformly identify the need for corrective action, control limits for equipment and analytical results are stated throughout this program.

Each contractor is required to submit internal QC procedures which include control limits and corrective actions to be taken if these limits are exceeded. These control limits are stated for each measurement method and are presented in tabular form in Section 6, Sampling Internal QC Checks, and Section 7, Analytical Internal QC Checks, of this QA Plan. Each media sampled or analyzed is covered in a separate table for easy use by a field or laboratory technician.

11.2 Immediate Corrective Action

Equipment and instrument malfunctions are most amenable to on-the-spot, immediate corrective actions. The internal QC tables are working tools which identify appropriate immediate corrective actions to be taken when a control limit has been exceeded; they provide the framework for uniform actions as part of normal operating procedures. The actions taken should be noted in field or laboratory notebooks but no other formal documentation is required, unless further corrective action is necessary. These on-the-spot corrective actions are an everyday part of the QA/QC system; they help avoid the collection of poor quality data.

11.3 Long-Term Corrective Action

The need for this action may be identified by standard QC procedures, control charts, performance or system audits. Any quality problem which cannot be solved by immediate corrective action falls into the long-term category.

The essential steps in a corrective action system are:

- Identify and define the problem.

- Assign responsibility for investigating the problem.

- Investigate and determine the cause of the problem.

- Determine a corrective action to eliminate the problem.

- Assign and accept responsibility for implementing the corrective action.

- Establish effectiveness of the corrective action and implement it.

- Verify that the corrective action has eliminated the problem.

Documentation of the problem is important to the system. A Corrective Action Request Form is filled out by the person finding the quality problem. This form identifies the problem, possible causes and the person responsible for action on the problem. The responsible person may be an analyst, field team leader, project QA coordinator or the QA Manager. If no person is identified as responsible for action, the QA Manager investigates the situation and determines who is responsible in each case.

The Corrective Action Request Form (CA form) is shown in Figure 9 and includes a description of the corrective action planned, the date it was taken, and space for follow-up. The QA Manager checks to be sure that initial action has been taken and appears effective and, at appropriate later dates, checks again to see if the problem has been fully solved. The QA Manager receives a copy of all CA forms and enters them in a Corrective Action Log. This permanent record documents follow-up and makes any quality problems visible to management; the log may also prove valuable for similar problems and their solutions.

The QA Manager maintains an active follow-up file, filing the CA forms in date order. If the follow-up on the indicated date shows a need for other action or continued follow-up, the action to be taken is identified on the form and it is filed under the date for the next follow-up. This system has proved quite effective in handling sequential types of corrective action since it brings the CA form to the QA Manager's attention at a time appropriate to check on the next stage of corrective action. The same form can follow a problem until it has been solved.

Contractors are required to have a functioning system which ensures that quality problems are corrected and follow-up is provided to

Originator

Date

Person Responsible for Replying

Contract Involved

Description of problem and when identified:

State cause of problem, if known or suspected:

Sequence of Corrective Action: (If no responsible person is identified, bring this form directly to QA Manager.)

State Date, Person, and Action Planned:

CA Initially Approved By:

Date:

Follow-up Dates

Final CA Approval By:

Date:

Information copies to:

RESPONSIBLE PERSON:

QA MANAGER:

DEPARTMENT MANAGER:

Figure 9. Corrective Action Request Form.

prevent recurrence of the problem. Contractors shall describe their internal system, how it operates in their specific project activity, and the person responsible for corrective action at each stage in their operations. They shall maintain a corrective action log for this project and include it in the project document inventory returned to EPA on completion of project work.

APPENDIX 1

SUGGESTED FORMAT FOR ANALYTICAL SUBCONTRACTORS' QUALITY ASSURANCE PLANS

SUGGESTED FORMAT FOR ANALYTICAL SUBCONTRACTORS' QUALITY ASSURANCE PLANS

The Quality Assurance Plans prepared by the contractors should be in formats consistent with each other as well as with the overall program QA Plan such that the Contractors' Plans can be incorporated into the Dallas Lead Quality Assurance plan as appendices. Therefore, it is recommended that the contractor follow the format listed below:

1. Outline for overall QA Project Plan, addressing each topic as it refers to your specific operation. See attached sample outline for contractor's QA Plan.

2. Document Control heading (on upper right-hand corner of each page). These may be used as four-line headings, if preferred.

EXAMPLE 1:	Appendix B, Section 1.1.1
(3-line)	Revision 0
	Page of
EXAMPLE 2:	Appendix Q13, Section 1.1.1
(4-line)	Revision 0
	Date
	Page of

CONTRACTOR'S QA PROJECT PLANS FOR ANALYSIS OF SAMPLES FROM DALLAS TEXAS

Outline

1.0 Project Description and Organization

2.0 Major Project Elements

3.0 QA Objectives - State expected precision and accuracy to be achieved by each analytical method in each media analyzed.

4.0 Document Control Procedures - Overall framework, numbering system, sample tags, Custody Forms, Log Books, Data Sheets provided by Prime Contractor. State your internal operation of the system, identifying responsible person as Document Control Officer.

5.0 Chain of Custody - Describe operating and security procedures. Describe procedures for sample receiving, sample storage, sample tracking, sample preparation, sample analysis, recordkeeping, data review and data reporting.

6.0 Analytical Internal QC Checks - Provide a table for each media (soil, dust, etc.) analyzed which lists operational checks for each analytical method. Control limits for each operational check should be provided with the corrective action to be taken if control limits are exceeded.

7.0 Data Reporting Management System - Standardized data forms will be provided by Prime Contractor - both analytical data sheets and encoding forms for the data management system. Internal operation of the system, and data review before reporting should be described.

8.0 Corrective Action Procedures - State how corrective actions will be implemented in the laboratory - identify responsible personnel. The corrective actions should have been identified in Section 6.0.

Appendix B Analytical Procedures

Part 1. Analytical Methods. Provided by EPA.

Includes analytical data sheets. Calibration of sampling equipment forms part of operating procedure--subcontractor provides for this equipment, unless given in analytical method.

Part 2. Analytical Equipment Maintenance/Troubleshooting Procedures

Analytical Balances
AA - Techniques
pH and Selective Meters

APPENDIX D

ATTACHMENT 1

DALLAS LEAD STUDY SOIL SAMPLING AND SAMPLE BANK PROCEDURES PROTOCOL

July 7, 1982

Soil Sampling and Sample Bank Procedures Revisions

Date revised - July 9, 1982

1. Attachment 2, page 7 of 9. e. Blood Samples (1) Blood Samples will be assigned sample bank numbers.

   e. Blood Samples (1) line 5. Analytical tags will be used.

   e. Blood Samples (3) Data reporting forms will not be used for blood samples. E.S.A. (E.S.A. Laboratories, Inc.) is providing laboratory coding forms.

2. Attachment 2, page 8 of 9. g. Quality Control Samples. (5) The sample bank personnel will generate one sample bank blank after preparing 40 soil samples.

Date revised - July 15, 1982

1. Attachment 2, page 4 of 9. (7) Last paragraph, Quality Control samples shall not be entered on the data reporting forms. All rows on the data reporting form wil be used for dust sample entries.

Date revised - August 17, 1982

1. Attachment 2, page 8 of 9. g. Quality Control Samples. (4) Four rows on each data reporting form will not be left blank. All rows on the data reporting forms will be used for soil and dust sample entries. Quality Control samples identified as QB0001, QS0001 and QA0001 will be reported on soil and dust sample quality assurance forms. See Analytical Protocol, Attachment 3, Section 1, pages 17 of 19, 18 of 19, and 19 of 19.

Date revised - September 1, 1982

1. Attachment 2, page 9 of 9. (7) Split samples with identification numbers ending with the capital letter X will be sent to:

Dr. Steve J. Simon
U.S. EPA
Environmental Monitoring Systems Laboratory
P.O. Box 15027
Las Vegas, Nevada 89114

DALLAS LEAD INVESTIGATION

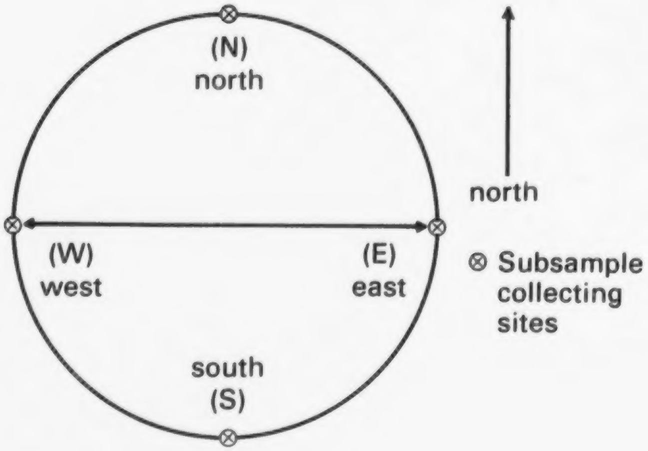
SOIL SAMPLING PROTOCOL

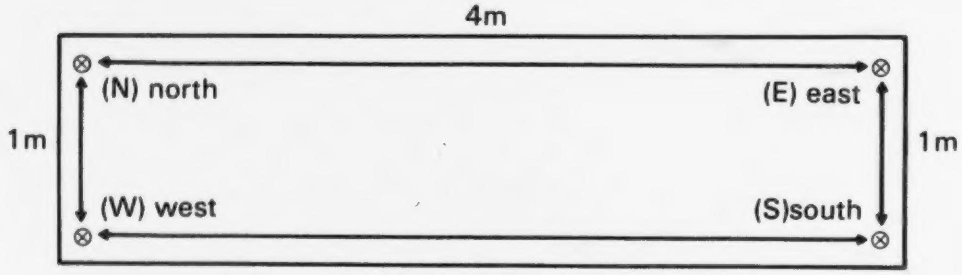
A. Location

1. U.S. Environmental Protection Agency (EPA) personnel from the Environmental Monitoring Systems Laboratory at Las Vegas (EMSL-LV) and/or the U.S. Public Health Service (PHS) personnel from the Center for Disease Control (CDC) at Atlanta will designate and identify all sampling locations.

2. Samples will be obtained by compositing subsamples of approximately 40 g each collected from four (4) equally spaced locations on the arc of a ten (10) meter diameter circle. One subsample will be collected at the northernmost point on the arc, one at the southernmost point, one at the easternmost point and one at the westernmost point.

Some of the designated soil sampling locations will not be large enough in area to collect soils around the required ten (10) meter diameter circle. The reduction of the diameter of the circle by one (1) meter increments to a minimum circle diameter of three (3) meters will be acceptable. If a sampling circle, as illustrated below, is not possible, measure and collect four (4) subsamples from a rectangular configuration as shown in the second diagram.





Soil collected from the four subsampling sites, located in each corner of the rectangle will be composited identical to those collected from the sampling circle.

3. Two methods of locating soil sampling locations will be used:

(1) A square sampling grid measuring 750' by 750' will be used to select sampling locations within and covering a 1 mile radius from both the RSR Corporation (RSR) and Dixie Metal Company (DMC) lead smelter sites. The grid will also be used to identify sampling locations at a reference (REF) site. The dimensions of the grid at the REF site will be identical to that used at the RSR and DMC sites. The area sampled at the REF site will only include a one-half mile radius from the intersection of Frio Drive and Kiest Boulevard.

The axes of the grid at the RSR, DMC and REF sites will run north-south, and east-west. The exact sampling locations will be identified on aerial photographs for each of the three areas. Samples will be collected near each grid intersection via the circular or rectangular configurations previously described.

(2) Specific locations at schools, parks, playgrounds, and/or in yards will be selected for sampling. These sampling locations will be selected by CDC personnel on a house-by-house and location-by-location basis to complement blood lead data. The sampling locations will primarily be contingent upon the activity patterns of preschool-aged children. Other criteria used to select soil sampling locations will be identified by CDC personnel. Soil samples will be collected at each of these locations via the circular or rectangular configurations previously described.

B. Frequency

1. A total of 177 soil sampling locations will be selected from the 750' by 750' grid intersections at each of the two (RSR and DMC sites. Approximately 89 sampling locations will be selected at the REF site.

Approximately 1,200 additional sampling locations will be selected at specific homes, parks, schools and playgrounds. At five (5) percent of the sampling locations duplicate samples will be collected. At an additional five (5) percent of the sampling locations the samples collected will be split into three homogeneous portions. At the duplicate sampling locations, the duplicate subsamples will be collected within a 0.5 meter radius of the original subsample collection location. The duplicate subsamples will be composited separately and prepared identical to those collected at the original subsite locations.

C. Pollutant Class

The soil samples will be collected for the analysis of lead.

D. Field Blanks

Field blanks are used to monitor sample handling procedures, container cleanup, and to document cross contamination. Field blanks are to be prepared in the field and handled in the same manner as collected samples. Field blanks will be placed and remain in the field sample storage container during the daily field sampling effort.

The field blank will be analyzed by the laboratory analyzing the samples, therefore, a field blank is required for each type of sample collected and for each media (soil and dust) collected. Unless otherwise stated, the field blank will be DDI (distilled deionized) water. The sampling teams as selected by sample bank personnel shall transport DDI water to the sampling locations and prepare one field blank for every 20 soil samples collected. The field blank will be DDI water that has come in contact with the soil corer.

E. Sampling Procedures

1. Container

The soil samples are to be contained in sealable polyethylene containers that have the capacity to hold 200 g. The samples are to be kept in a covered container at ambient meteorological conditions until delivered to the sample bank.

2. Container Preparation

The containers will be sterile 27 ounce whirl-pak polyethylene containers. As such, precleaning will not be required. These sample containers should be stored and kept in a closed container to minimize atmospheric contamination.

3. Sample Collection and Handling Procedures

A nickel chrome-plated soil corer with an inside diameter of 0.75 inches, and capable of a vertical penetration into mineral soil 3 inches deep, shall be used to extract the four 3-inch deep core subsamples. The composite samples will consist of all four subsamples placed in the same sample container.

The sample shall consist only of mineral soil. If sod layers are encountered, such as grass, remove the surface vegetative sod material by dissection using a scalpel. Discard the scalpel blade and replace with a clean blade after sampling at all four subsites. Pre-cleaned polyethylene gloves must be worn by the sampler to avoid sample contamination during collection and/or dissection.

Place any vegetative material in a clean polyethylene container and attach a properly filled out vegetative collection tag to the container. Vegetative subsamples will be composited identical to soil subsamples.

The senior member of the field sampling team will be responsible for adhering to the following sample collecting guidelines.

1. At designated grid sampling locations the sampling site configuration (circle or rectangle) shall be located at the exact grid intersection point. If this location is unsatisfactory due to streets, structures, and/or other obstructions, the sampling site can be moved within a 30-meter radius of the grid intersection. If a sample cannot be obtained within the 30-meter radius the on-scene coordinator should be notified.

2. Avoid if possible collecting samples that are less than 20 feet from painted surfaces.

3. Locate collection sites as far as possible from vehicle activity such as streets, driveways, parking and automobile repair areas.

4. Avoid if possible collecting samples under or immediately adjacent to trees, shrubs, and/or structures.

5. Complete site description forms (See Part F).

6. Thoroughly wash and rinse the soil corer in hot tap water and then rinse the corer with DDI water after sampling at each location. A suitable brush or Kimwipes may be necessary for adequate cleanings. If hot tap water is unavailable, use cold tap water with a suitable lead-free laboratory detergent. Label the sample container with an appropriate sample collection tag (as shown) supplied by the sample bank.

<table>
<tr><td>Project Code
Dallas Lead</td><td>Soil</td><td>(Source of Sample)
Grid Park School
Home Playground Other</td><td colspan="3">(Site Code)</td><td rowspan="4">Tag No.</td></tr>
<tr><td colspan="3">Address of Site (or Location)</td><td>Date</td><td colspan="2">Time
(Military)</td></tr>
<tr><td colspan="6">Sampler (signature)</td></tr>
<tr><td colspan="4">Remarks:

Sample I. D. No. ____________________</td><td>Lead
XXXX</td><td>Analysis</td></tr>
</table>

Sample Collection Tag

The sample collection tags have been designed for soil, vegetation and dust samples. They are a different color for each of the three media and are serially numbered starting with a different block of numbers for each media and for each (RSR, DMC and REF) site. The tags are preprinted to ensure that the required information is provided on each tag. Each collected sample, including duplicates and field blanks, shall have a completely filled-in sample tag securely attached to it. Duplicates and field blanks shall be identified in the "remarks" section of the tag and by a specific sampling site identification code number. See Site I. D. Soil Grid Sampling Sites, Attachment 1. Samples collected for splits shall be identified in the remarks section of the tag.

The person who physically collects the sample is the SAMPLER and signs the sample tag. The SAMPLER initiates the custody record for transfer of samples to the sample bank.

After the samples have been transferred to the Sample Bank and documented there, they will be renumbered with a sample bank number and identified with a new sample analysis tag as shown below. This renumbering step enables the submittal of duplicate samples and QC samples as blinds to the analytical laboratory.

(Project Code) Dallas Lead	(Sample Bank No.)
(Sample Description)	
Analysis	
Metals (Lead)	
(Tag No.)	

Analysis Tag

The original sample collection tag number is recorded in the sample bank's master log and the tag itself is stored in the document file at the Sample Bank (see Attachment 2, Sample Bank Operation). For vegetation samples only leave the sample collection tag on the sample container. Traceability of samples from collection location to laboratory analysis is required as such, cross indexing of all sample collection tag numbers, and corresponding sample bank and analytical tag numbers must be made in the master logbook.

The sample bank will also be responsible for drying and mixing each soil sample. This drying and mixing procedure to achieve homogeneity will be accomplished by placing the total soil sample in a clean 250 ml glass beaker. Place the beaker in a drying oven at a temperature of 100°C for an 8-hour period or until the soil sample is dry. After drying, place the soil in an 8" diameter stainless steel 16 mesh sieve. Sieve the soil through the sieve with the aid of a glass pestle. Discard the unsieved portion. Place the sieved portion in a lead-free porcelain grinding jar and mix by placing the jar on a multi-tiered jar mill and rotate at a speed of 250 rpm for a 30-minute period. After the 30 minute mixing period place 1/3 or approximately 50 g of the dry soil in a clean polyethylene sample container and attach a properly filled out sample analysis tag. The remaining 2/3 of the soil sample shall be placed in a clean polyethylene sample container and properly labelled so that it can be identified as being part of the same sample as the analyzed (1/3) portion. The 2/3 portion shall be stored at the sample bank.

The vegetative samples shall be placed in a freezer at 0°C and stored at the sample bank. Analysis of the vegetative tissue may be required at a later date. Those soil samples identified as splits on the sample collection tag will be dried and mixed and then split into three equal but separate samples for analysis. One of the split samples will be shipped to the referee analytical laboratory. The remaining two split samples will be shipped to the same analytical laboratory. The identification of these split samples must be documented in the master log. Samples identified as duplicates on the sample collection tag will also be shipped to the same analytical laboratory.

The equipment used for drying and mixing the soil must be decontaminated after each sample. Wash and rinse each component of the drying and mixing equipment in hot tap water and then rinse with DDI water. Use a lead-free laboratory detergent and laboratory brushes or Kimwipes in the decontamination procedure if required.

F. Site Description Form

An example of the site description form that will be used in this study is shown as Figure 1. The senior member of the field sampling team will be responsible for completing one site description form for each location sampled. Documentation at each site should include appropriate photographs showing the area sampled. In addition, a schematic drawing showing sampling locations, including dimensions and other pertinent information such as structures, ground surface characteristics, streets and driveways will be prepared.

The site description forms will be deposited and stored at the sample bank.

G. Sample Transfer Procedures/Chain-of-Custody

There will be a number of transfers of custody in this program. Samples are to be delivered to the sample bank by the sampler. The samples will be shipped to analytical and referee laboratories by the sample bank. In addition Quality Control (QC) samples, provided by other laboratories will be shipped to the sample bank for inclusion with collected environmental (soil, vegetation, dust and blood) samples. All entities that generate samples for this study are required to initiate custody records.

Customized chain-of-custody record sheets, following the NEIC format will be provided by the sample bank. Examples of these record sheets are shown as Figures 2 through 4. Figure 2 is used for samples collected in the field, Figure 3 is for shipping QC samples to the sample bank and Figure 4 is used for shipping samples to analytical laboratories.

The custody record sheets will be used for a packaged lot of samples; more than one sample will usually be recorded on one form. More than one custody record sheet may be used for one package, if necessary. Their purpose is to document the transfer of a group of samples traveling together; when the group of samples changes, a new custody record is initiated. The original of the custody record always travels with the samples; the initiator of the record keeps a copy. When custody of the same group of samples changes hands several times, some people will not have a copy of the custody record. This is acceptable as long as the original custody record shows that each person who had received custody has properly relinquished it.

Using the Custody Record Sheet

- The originator fills in all requested information from the sample tags.
- The person receiving custody checks the sample tag information against the custody record. He also checks sample condition and notes anything unusual under "Remarks" on the custody form.
- The orginator signs in the top left "Relinquished by" box and keeps the copy.
- The person receiving custody signs in the adjacent "Received by" box and keeps the original.
- The Date/Time will be the same for both signatures since custody must be transferred to another person.
- When custody is transferred to the Sample Bank or an analytical laboratory, blank signature spaces may be left and the customized last "Received by" signature box used.
- In all cases, it must be readily seen that the same person receiving custody has relinquished it to the next custodian.
- If samples are left unattended or a person refuses to sign, this must be documented and explained on the custody record.

The sample bank will retain copies and/or original shipping papers, bill of lading and other records dealing with the shipment of samples.

If discrepancies between sample tag numbers and custody record listings are found, the person receiving custody should document this and properly store the samples. The samples should not be analyzed until the problem is resolved by contacting the on-scene coordinator or other designated responsible authority.

The responsible person receiving custody should attempt to resolve the problem by checking all available information (other markings on sample container, type of sample, etc.). He should then document the situation on the custody record in his project logbook and notify the on-scene coordinator as soon as possible.

Changes may be written in the "Remarks" section of the Custody record and should be initialed and dated. The copy of this record should accompany the written notification to the on-scene coordinator.

Custody Seals

Custody seals will be required when shipping samples by commercial carriers. The seals are narrow strips of adhesive paper used to demonstrate that no tampering has occurred. They are intended for use on a sample transport container which is not secured by a padlock. They are not intended for use on individual sample containers.

Custody of Shipped Samples

When a group of samples with its custody form is to be shipped, the shipper (e.g. sample bank personnel) accompanies the package to the carrier (e.g., Federal Express) so that, if requested, the number and identification of the samples in the container can be verified. The commercial carrier is not required to verify this.

The package is then closed with a strong, keyed padlock or strapping tape and custody seals so that the carrier is transporting a secure container.

NEIC's recommended procedure for custody seal use on shipping boxes is as follows:

- Place paper side of seal against box and wrap strapping tape across the middle of the seal and at least twice around the box.
- Cut the tape so that the ends overlap at the seal.
- Fold the seal to itself over this overlap and sign the custody seal.
- Repeat this procedure as many times as necessary to seal the box for shipment.

The person receiving custody of shipped samples must document the condition of the locked, or strapped and sealed shipping box on arrival. It must be checked that neither the tape or the custody seals has been cut or otherwise tampered with. If the paper seal has been damaged in shipping but it is clear that the shipping box has not been opened, further handling of the samples may proceed. If tampering is suspected, the designated Sample Custodian shall notify the on-scene coordinator and the sample bank supervisor.

PROJECT CODE: Dallas Lead. DATE ____________

SAMPLER: ____________________

SITE ADDRESS: (STREET, NUMBER, COORDINATES) ______________________________

TYPE OF SITE: (RESIDENTIAL, INDUSTRIAL, ETC.) ______________________________

PROPERTY OWNER: ______________________________

LOCATION OF SAMPLING: (BACKYARD, PARK, ETC.) ______________________________

*SKETCH MAP OF THE SAMPLING AREA AND ATTACH IT TO THIS FORM.

ATTACH POLAROID PHOTOGRAPH OF SAMPLING SITE TO THIS FORM.

ADJOINING PROPERTY: (RESIDENTIAL, INDUSTRIAL, ETC.) ______________________________

GROUND SURFACE: (BARE, LAWN, ETC.) ______________________________

STRUCTURAL ODDITIES: (SWIMMING POOLS, SHEDS, ETC.) ______________________________

TYPE OF SAMPLING PERFORMED: (SOIL, DUST, ETC.) ______________________________

SAMPLE COLLECTION TAG NUMBERS: ______________________________

CONDITION OF SAMPLING SITE: (DEBRIS, RESIDUES, STANDING WATER, ETC.) ______________________________

REMARKS: (INCLUDING ALL SAMPLE NUMBERS) ______________________________

*Sketch of the sampling area should be prepared with fixed landmarks suitable as reference locations. Distances and locations must be measured by tape or hand-held range finding equipment, or a combination of both.

Figure 1. Sampling Site Description Form.

Figure 2.

Environmental Protection Agency
Office of Enforcement

CHAIN OF CUSTODY RECORD

Proj. No.

Project Name DALLAS LEAD

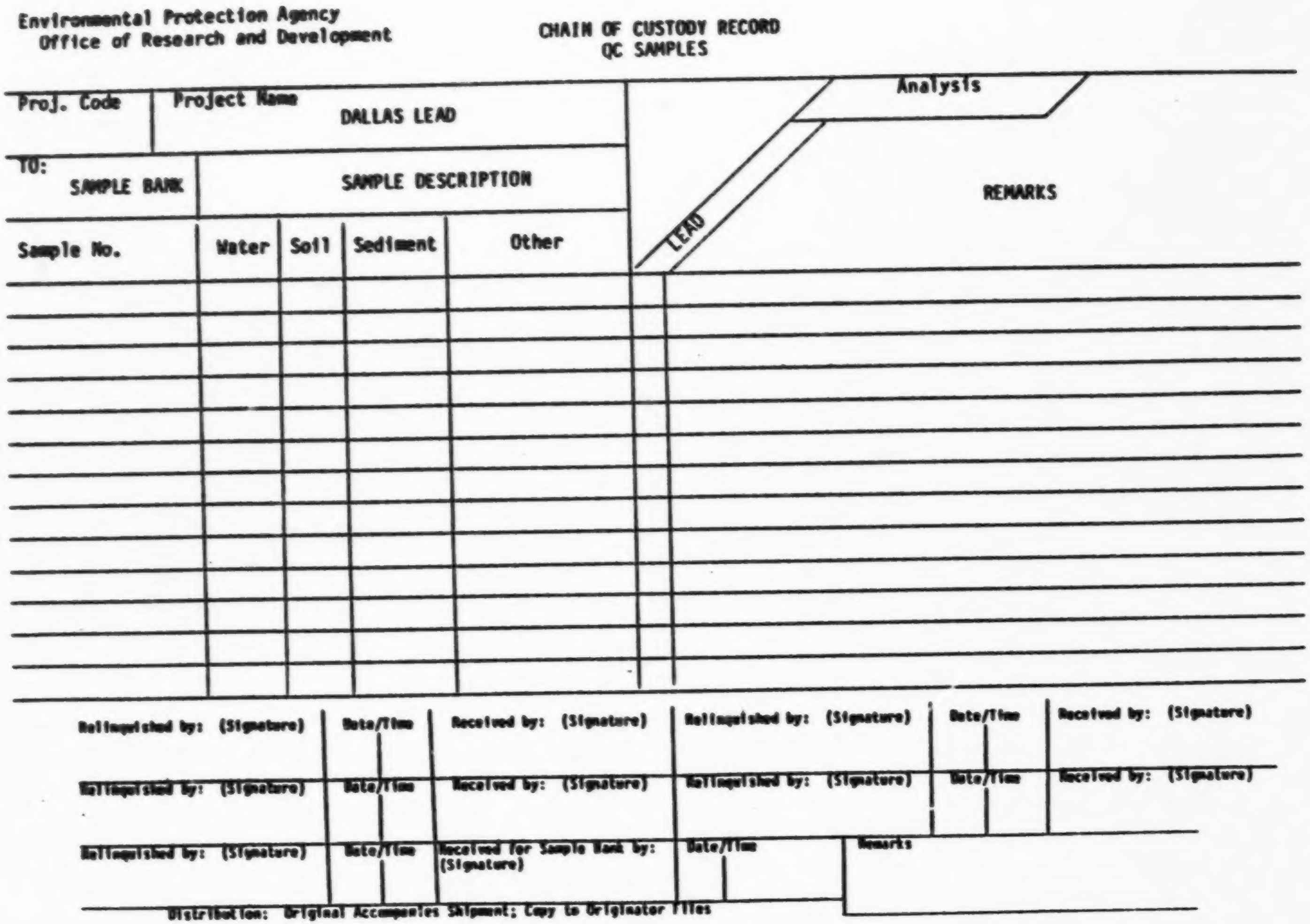
SAMPLERS: (Signature)

							Analysis		Sample Type					
TAG NO.	DATE	TIME	COMP.	GRAB	SITE LOCATION	No. of Containers	LEAD		SOIL	DUST	BLOOD	VEGETATION	OTHER	REMARKS

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Sample Bank by: (Signature)	Date/Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Originator Files

Figure 3.



Environmental Protection Agency
Office of Research and Development

CHAIN OF CUSTODY RECORD
QC SAMPLES

Proj. Code	Project Name DALLAS LEAD				Analysis	
TO: SAMPLE BANK	SAMPLE DESCRIPTION					
Sample No.	Water	Soil	Sediment	Other	LEAD	REMARKS

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Sample Bank by: (Signature)	Date/Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Originator Files

Figure 4.

Environmental Protection Agency
Office of Research and Development

CHAIN OF CUSTODY RECORD

Proj. Code	Project Name DALLAS LEAD				Analysis	
TO: ANALYTICAL LAB	SAMPLE DESCRIPTION					REMARKS
Sample No.	Water	Soil	Sediment	Other	LEAD	

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Laboratory: (Signature)	Date/Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

ATTACHMENT 1

SITE I.D. SOIL GRID SAMPLING SITES

I. Sampling Site Identification (Site Code)

Aerial photographs of each area (RSR, DMC and REF) overlayed by a 750' x 750' soil sampling grid will be provided to each sampling team. Every grid line is identified. The rows are identified alphabetically, and the columns numerically.

Each soil sample collected from the grid configuration will have a site identification code that will identify the grid site from which the sample was collected. It is required that all forms that originate with the sample collection (sample collection tags, site description forms and chain-of-custody forms) have a site identification code on them. Sample bank personnel will not accept any sample in which information, as required on the forms, is missing. The sampling team leader will be responsible for making sure all required sampling forms are properly filled out.

The grid sampling site identification code will be as follows:

a. The first three letters will identify the grid location, RSR, DMC or REF.

b. Place a hyphen - after the last letter of the grid location designation.

c. The fifth space will be a letter identifying the row (A through O).

d. The sixth and seventh spaces will be numbers identifying the columns (01 through 15).

e. A typical site identification code should appear as RSR-F08. At five percent (5%) of the grid sampling sites (listed below) duplicate samples will be collected. The duplicate (the second one collected) will be identified by adding an A immediately following the number identifying the column. For example:

   RSR-E03 identifies the first soil sample collected at the RSR location, at the grid intersection located in row E, column 03, while RSR-E03A identifies the duplicate sample collected at the same location, row and column.

Duplicate soil samples will be collected at the following row and column grid sampling sites: E03, E14, F12, G02, G15, H14, I05, I06, J11, K04. For every 20 soil samples collected from the grid sampling sites one field blank will be collected. The sample bank personnel will select and notify the team responsible for collecting the field blank on a daily basis as required. Field blanks will be identified with a specific soil sampling site. The field blanks will be collected immediately prior to collecting a soil sample at a designated site. For example, if the next soil sampling site is identified as RSR-F08, prepare the field blank first before collecting the soil sample. Identify the field blank by placing a B immediately following the number identifying the column; i.e., RSR-F08B.

The appropriate site identification code for each soil sample collected by grid designation including duplicate and field blanks must appear on the sample collection tag in the space identified as (site code) on the chain-of-custody form space identified as site location, and on the site description form space identified by site address.

Designations X, Y and Z immediately following the column identification number will indicate the soil sample as a split: i.e., RSR-D02X, RSR-D02Y and RSR-D02Z. The sample bank personnel however, will add this designation. Soil samples designated for splitting will be collected at the following row and column grid sampling sites: D02, E07, E09, F03, J07, K09, L06, M10, M13, N05.

The sampling teams will also be provided aerial photographs of each location that will enable the sampling team leader to plot and identify the exact sampling site.

ATTACHMENT 2

SAMPLE BANK PROCEDURES

I. Responsibility

The sample bank is the custodian for all records pertaining to the sampling, sample preparation as required, and shipment of environmental samples to analytical laboratories.

The sample bank is responsible for record filing and storing, for storing and preparation of soil and vegetation samples, and for dispensing containers, sampling equipment and all custody documents such as chain-of-custody forms and sample collection and analytical tags, as required.

The sample bank is responsible for updating and maintaining the projects' master log book, auditing the records as required, generating sample bank QC sample blanks, accepting QA/QC samples for inclusion into the analytical scheme, and for scheduling the collection of field sample blanks.

The sample bank is responsible for completing, as required, analysis data reporting forms and for assuring that all chain-of-custody requirements as stated in the Dallas Lead Studies Quality Assurance Plan, pertaining to all field sampling, shipping and sample bank operations, are adhered to.

II. Procedures

a. Issuing Supplies:

(1) On a daily basis, or as appropriate, the sample bank will issue as required sample containers, sample collection tags, (soil, dust, blood, and vegetation), chain-of-custody forms as shown on Figure 2, and site description forms to the sampling teams. As the sample collection tags and chain-of-custody forms are accountable documents, the sample bank will log the forms by numerical lot identifying the team and/or the individual responsible for the temporary custody of these documents.

(2) The sample bank may be required to store sampling equipment in a suitable environment as stated in Section 5.0 of the Dallas Lead Quality Assurance Plan. If sampling equipment is stored at the sample bank, issuing this equipment to the sampling teams on a daily basis or as required, will be necessary.

b. Accepting and Logging Samples:

(1) Transfer of sample custody from the sampler to sample bank personnel (any sample bank personnel may accept custody) will normally occur at the sample bank.

(2) Before accepting custody of any samples, sample bank personnel must check all tags and forms for legibility and completeness.

(a) All individual samples must have a completely filled out sample collection tag attached.

(b) Every sample must be identified on the chain-of-custody form.

(c) Each site sampled must have a completely filled out site description form.

(d) Any discrepancy will be corrected before the sample bank personnel will assume custody. If a discrepancy exists that cannot be resolved to the satisfaction of the sample bank personnel, re-sampling, filling out additional tags and forms, and/or re-visiting the site to obtain necessary documentation may be required.

(e) All unused accountable documents must be returned to the sample bank on a daily basis. However, depending upon circumstances such as a sampling team's schedule and route, accountable documents may be retained by the sampling team leader. The sample bank supervisor, however, must be aware of the situation.

(3) After the sampler relinquishes custody and the sample bank personnel assumes custody of the samples, each sample must be logged into the master log book. The master log book should be a suitable ledger with column headings, as shown on Figure 5. Additional headings may be required as the program develops, as such, blank columns should be available in the ledger.

c. Dust Samples:

(1) All sample bank numbers designating dust samples should begin with the capital letter D. The remaining five digits will be numbers beginning with 00001., i.e., the first dust sample logged into the log book will be D00001. The second sample D00002, etc.

Sample Bank No.	Sample Collection Tag No.	Date Collected	Media	Chain-of-Custody Form No.	Site I.D. Code No.	Link No.	Address	Analytical Tag No.	Analytical Laboratory (Initial)	Date Sent to Analytical Laboratory	Chain-of-Custody Form No. to Analytical Laboratory	Remarks
D00001	15031	4 July 82	Dust	05301	RSR-01		109 1st Street	00231	GCA	7 July 82	05490	

Figure 5. Master Log Book Headings.

(2) After assigning and logging the sample bank number, record in the log book the sample collection tag number, link number (No. on computerized preprinted tag), the date the sample was collected, the media (dust), the chain-of-custody form number that the sample appeared on when custody of the sample was assumed by the sample bank, the site I.D. Code and the address where the sample was collected.

(3) Remove the sample collection tag from the sample, attach to the sample an analytical tag, and record the analytical tag number in the log book. Record the sample bank number and the sample description (dust) on the analytical tag. The sample is now ready for shipment or it can be stored until additional samples are logged.

(4) When the samples are prepared for shipment, record in the log book the initials of the analytical laboratory to which the sample is to be sent and the date the sample was shipped.

(5) Fill out a chain-of-custody form (Figure 4), identifying the analytical laboratory to which the samples are to be sent, the sample bank number of each sample in the shipment, the sample description (dust), and any remarks concerning the sample that are appropriate. Record the chain-of-custody form number in the log book.

(6) Remarks concerning the condition of a sample or other pertinent sample information can be beneficial, especially during data interpretation. Record in the log book any information that may be appropriate.

(7) Fill out two analysis data reporting forms (Figure 3, Section 2.0 in Quality Assurance Plan Dallas Lead Study). One form will be included with the sample shipment to the analytical laboratory. Record all available information on this data reporting form except the Sample I.D. Code number and/or site address.

Each sample shipped must be listed on the data reporting form before shipping.

Leave four rows blank on each data reporting form used. These rows will be used for Quality Control samples (See Section 3, Quality Control samples) before sample shipment. The samples can be packaged and shipped as previously identified in the soil sampling protocol.

The second data reporting form will have all available sample information recorded. Include the Site I.D. Code number and/or address on this form.

(8) Make two copies of the second data reporting form, check for legibility. Send one copy to: (RTI)

Mr. Robert M. Lucas
Research Triangle Institute
P.O. Box 12194
Research Triangle Park
North Carolina 27709

and the other copy to: (EMSL-LV)

Dr. Stuart C. Black
U.S. Environmental Protection Agency EMSL-LV
P.O. Box 15027
Las Vegas, NV 89114

(9) The following documentation and forms and their disposition for each sample (i.e., D00001) from collection to the analytical laboratory is as follows:

Form	Disposition
1. Sample Collection Tag	Filed at Sample Bank
2. Site Description Form	Filed at Sample Bank
3. Sampler Chain-of-Custody Form	All copies filled at Sample Bank
4. Master Log Book	Sample Bank
5. Analytical Tag	Analytical Laboratory
6. Chain-of-Custody Form (to Analytical Laboratory)	Original to Analytical Laboratory Copy to Sample Bank
7. Data Reporting Form (with Site I.D. No.)	Original - Sample Bank Copy - RTI Copy - EMSL-LV
8. Data Reporting Form (without Site I.D. No.)	Original - Analytical Laboratory Copy - Sample Bank
9. QC Chain-of-Custody Form	Original - Sample Bank Copy - Originator of QC Sample
10. Shipping Form	Original - Analytical Laboratory Copy - Sample Bank

d. Soil Samples:

(1) All sample bank numbers designating soil samples should begin with the capital letter S. The reamining five digits will be numbers beginning with 00001, i.e., the first soil sample logged into the log book will be S00001, the second soil sample S00002 and etc.

(2) The log book should be arranged in such a manner as to log media in groups. For example, all soils should be logged together, all dust together and etc.

(3) Logging of soil samples will be identical to that described for dust samples. The soil samples, however, must be prepared for analysis by sample bank personnel before they can be shipped to the analytical laboratory. The sample should not be given an analytical tag until after preparation.

(4) After assigning a sample bank number to the soil sample and recording the proper information into the log book, place the soil sample into a properly identified drying container (label the container with the sample bank number). Follow the soil sample preparation procedures as identified in the soil sampling protocol.

(5) If the sample is a split as previously identified in Attachment 1 (those samples collected from row and column grid sampling sites D02, E07, E09, F03, J07, K09, L06, M10, M13, and N05) divide the sample after rotating (mixing) into three equal portions. Package each of the three portions into clean polyethylene containers, attach a completely filled out analysis tag to each sample, assign a sample bank number to the two additional soil samples, place an X, Y and Z designation at the end of the site identification code numbers and log all information into the log book.

(6) All soil samples are to be prepared identically, i.e. drying, sieving and mixing. For those soil samples that are not designated as splits, place one-third (1/3) of the mixed soil in a clean polyethylene container, attach an analysis tag to this sample and record the necessary information into the log book. The remaining two-thirds portion should be repackaged, labeled and identified by a sample bank number and then stored in a secure environment at the sample bank.

(7) Material that is discarded during the sieving procedure must be described, i.e. approximate amount, presence of paint chips and etc. Record this information in the master log book under remarks.

(8) The soil samples can now be prepared for shipment as previously described for dust samples. All documentation and forms used for dust samples are to be used for soil samples.

e. Blood Samples:

(1) Blood samples will not be assigned sample bank numbers. Record and log the link number (number attached to the blood sampling tube) into the sample bank log book. Link numbers will be used to identify blood samples at the analytical laboratory. Analytical tags will not be used. All other sample bank logging documentation and shipping procedure will be similar to that described for dust samples.

(2) Blood samples should be refrigerated while at the sample bank.

(3) Data Reporting forms should not be sent to Dr. Black at EMSL-LV.

f. Vegetation Samples:

(1) All sample bank numbers designating vegetation samples should begin with the capital letter V. The remaining five digits will be numbers beginning with 00001., i.e. the first vegetation sample logged into the log book will be V00001, the second vegetation sample V00002 and etc.

(2) After assigning the sample bank number, record the sample bank number, collection tag number, date collected, media, chain-of-custody form number, and the site I.D. Code number in the log book.

(3) Record the sample bank number in the remarks section on the sample collection tag. Don't remove the sample collection tag from the sample container; store the Vegetation sample as described in the soil sampling protocol.

g. Quality Control Samples:

(1) Quality control samples will be sent to the sample bank accompanied by chain-of-custody forms as shown on Figure 3. These samples will be appropriately identified by sample description and concentration. Additional information may be written in the remarks section of the chain-of-custody form.

(2) These QC samples will be given a sample bank number beginning with the capital letter N. The remaining five digits will be numbers beginning with 00001, i.e. the first Quality Control sample logged into the log book will be N00001, the second N00002 and etc.

(3) These QC samples will be logged in, documented, and shipped identically to procedures previously identified for dust samples.

(4) As previously mentioned, four rows on each data reporting form will be left blank. These rows are for recording data and information concerning analytical laboratory generated QC samples.

The analytical laboratory will fill in the required analytical information on the Data Reporting Form. The sample bank, however, will fill in the sample bank number on the form as follows:

QB0001 will denote a reagent blank
QS0001 will denote a laboratory control standard
QA0001 will denote a split extract

These sample bank numbers will be logged into the log book in the QC section. Also, these sample bank numbers should be logged in numerical order by category, i.e QB0001, QB0002, etc.

(5) The sample bank personnel will generate one sample bank blank after preparing 20 soil samples. The sample bank blank will be DDI water that has come in contact with both the soil sieve and mixing jar.

(6) These sample bank QC samples will be given a sample bank number beginning with the capital letter N followed by a capital letter B. The remaining four digits will be numbers beginning with 0001, i.e. the first sample bank blank logged into the

log book will be NB0001, the second NB0002 and etc. The sample bank blanks will be shipped to the analytical laboratory with the soil samples.

(7) Every soil sample designated as a split by the site identification code number ending with the capital letter X will be sent to

Dr. Ted Meiggs
USEPA
National Enforcement Investigation Center
Building 53, Box 25227
Denver Federal Center
Denver, Colorado 80225

The remaining splits (Y and Z designations) will be sent to the contractor laboratory.

(8) One of the two dust split extracts will be delivered to the sample bank for logging and shipment to Dr. Ted Meiggs.

(9) Duplicate samples designated by the capital letter A at the end of the site identification code will be sent to the same analytical laboratory.

(10) The following is a listing of sample bank identification numbers or link numbers and what they designate.

Number	Designation
D00001	Dust
S00001	Soil, Field Blanks
Link number	Blood
V00001	Vegetation
N00001	QC Samples sent to Sample Bank
QB0001	Reagent Blank
QS0001	Control Standard
QA0001	Split Extract
NB0001	Sample Bank Blanks

APPENDIX D

ATTACHMENT 2

DUST SAMPLING PROTOCOL

COLLECTION OF ENVIRONMENTAL DUST SAMPLE

Adverse health effects in children due to lead are generally due to ingestion of flakes or chips of lead-based paints. Blood lead levels lower than that found in lead poisoned children may be the result of ingestion or inhalation of materials containing lower concentrations of lead. Airborne lead from the combustion of leaded gasoline or stationary industrial sources contributed to lead bearing dust in the house, in the street, and in soil surrounding the dwelling.

For purposes of this study only dust inside the house and accessible to the child will be collected and assayed. Such materials may well contribute to the total body burden of lead. Dust which collects in all corners, on top of refrigerators, window and door facings is easiest to collect since larger quantities are available, but in this study only dust on the floor and easily available to the child will be collected.

The usual procedure of collecting samples with a small brush in a suitable container may not provide sufficient quantity for analysis. Therefore, we propose to wipe a surface with a suitable material and assay the dust collected. Either dry or wet wipes can be used, but wet wipes will collect more dust for the same amount of effort with less chance of losing some to a disturbed air flow. Individually packaged cotton swabs saturated with lead-free alcohol are convenient for the task.

Collection from one dwelling to the next should be systematic and as methodical as possible. If the area wiped is known, a potentially significant factor in the overall lead assessment is added. This can be calculated by a convenient pocket measuring tape, but a more reproducible method is to use a template with a known area cut from the center to delineate the area wiped.

Carry the 10 cm^2 template in the plastic bag provided for the purpose. Place the template on the kitchen floor in a corner. Always handle template by the outside, do not touch the inside edges. Put on the plastic gloves and prepare to open the individually packaged alcohol swabs (Becton-Dickinson No. 6894 or equivalent).

Before tearing open the package, press the swab from the top down, forcing the swab toward the bottom of the package away from the dotted line. Tear along the dotted line and discard the smaller portion of the package. Invert the package and squeeze all the alcohol possible from the package. The cotton swab will remain moist and this pressing will have the effect of making the swab somewhat fuzzy -- the better to collect dust particles.

Next, carefully tear the other two sides of the package apart, being careful not to touch the swab. The package is now completely opened and lying flat on the table. Open the second package in the same manner.

Grasp one corner of the swab, being careful to touch as little of the swab as possible, thence the opposite corner until you have the tips of each corner in hand. Place the pad thus formed in the upper right corner of the template and move to the left, moving in one direction only. When you come to the left side of the template, lift the pad, go back to the right side and repeat the procedure until the entire area within the template has been covered.

Place the pad with the dust covered side up on the used container that has been left lying conveniently nearby. Take the second alcohol swab by the corner as described and make a pad to wipe from top to bottom of the template. Begin in the top left corner and wipe in one direction toward the bottom of the template until the entire area has been covered the second time.

Place this pad directly on top of the first pad with the dust covered surface down. The two dust covered surfaces should now be facing each other on the open disposable swab container. Close the top and fold the container toward the closed side. Since we now have a foil package with two swabs that was designed for one, it will appear somewhat bulky and will try to unfold. A paper clip will suffice to keep it closed, but do not squeeze the package with sufficient force to expel the remaining alcohol for it may carry away the dust particles. Place in approved container with appropriate label.

Open a new swab and clean the inside of the template. Dry off with the cotton gauze pad and place in the transport container. This operation should have consumed no more than 5 to 10 minutes. Draw spot where soil was taken in the scheme on the environmental form.

Responsibility for the dust specimen thus collected rests with the environmentalist under terms of the chain-of-custody provided by the Environmental Protection Agency.

APPENDIX D

ATTACHMENT 3

DALLAS LEAD STUDY SOIL AND DUST ANALYTICAL PROTOCOL

June 30, 1982

Analytical Protocol Revisions

Date revised - August 27, 1982

1. Section 1, page 6 of 19, paragraph 1.4.7. Calibration standards of 0.10, 0.50, 1.0, 3.0 and 8.0 µg/ml Pb are prepared for the dust wipe analysis to reflect the generally low level of lead present.

2. Section 1, page 9 of 19, paragraph 1.6.1d. To each soil sample add 25 ml of D.I. water to form a slurry. To this slurry 25 ml [illegible] conc HNO_3 is slowly added. Recover the beaker with a watch glass.

3. Section 1, page 11 of 19, paragraph 1.6.2e. ...filter through Whatman No. 42 filter paper (or equivalent) into a 100 ml volumetric flask. Install lead hollow cathode lamp. Set the wavelength of the monochrometer at 283.3 nm for soil extract analysis and at 217.0 nm for the dust wipe analysis.

4. Section 1, page 14 of 19, paragraph 1.8.2. The minimum reportable level is 0.10 µg Pb/cm^2 based on the lowest working standard of 0.10 µg Pb/ml. Samples whose extracts are below 0.10 µg Pb/ml but are

above the limit of detection should be reported but the values should be bracketed ( ), ...

5. Section 2, page 1 of 22, paragraph 2.0. Under soil - as of 8/25/82 the reextraction of sample residue requirement (f) will be discontinued. Thus far, no detectable lead has been found in the sample residue.

6. Section 2, page 4 of 22, paragraph 2.1.2a. In order to verify the instrument calibration, a known control sample, such as the EPA Quality Assurance Water Samples - WPI (prepared by EMSL-CIN) or a contractor prepared standard which is independent of the calibration standards and prepared from NBS lead nitrate, will be analyzed.

   This change was instituted since available QA samples are too low in concentration even on a smaller dilution to serve as effective calibration control checks.

7. Section 2, page 6 of 22, paragraph 2.1.3. For every 40 samples collected in the field one soil container filled with D.I. water which was poured over a cleaned coring device will be sent ... For every 40 soil samples processed through the sample bank, D.I. water will be processed as above and through each sample bank operation and then ....

Date revised - September 14, 1982

1. Section 2, page 1 of 22, paragraph 2.0g. One "total recoverable" analysis (one per 40) where the parent sample is X9.

DALLAS LEAD SMELTER STUDY

Soil and Wipe Sample Analysis

1.0 Analysis of Samples

1.1 Apparatus

1.1.1 Glassware. Class A borosilicate glassware should be used throughout the analysis.

a. Beakers: 150 ml graduated Pyrex or Phillips beakers.

b. Volumetric flasks: 50 ml, 100 ml, 500 ml, and 1000 ml flasks.

c. Pipettes (to be calibrated at the beginning of the study).

d. Watch glasses.

e. Funnels.

f. Glass stir rods.

1.1.2 Hot plates and/or block digestors.

1.1.3 Drying oven.

1.1.4 Analytical balance capable of weighing to 0.1 mg.

1.1.5 Whatman 42 ashless filter paper, or equivalent.

1.1.6 Atomic Absorption Spectrophotometer equipped with lead hollow cathode lamp.

1.1.7 Acetylene: grade recommended by A.A. manufacturer.

1.1.8 Air: filtered to remove particulate, oil and water.

1.1.9 Mortar and pestle (agate or glass).

1.1.10 100-mesh sieve, U.S. standard or equivalent, with nylon or lead-free wire screen.

1.2 Reagents

1.2.1 Distilled-deionized water (D.I. water).

1.2.2 Concentrated nitric acid (HNO_3), ACS Reagent Grade or better.

1.2.3 NBS-SRM 928, Lead Nitrate ($Pb(NO_3)_2$) (see Appendix A).

1.2.4 NBS-SRM 1648, Urban Particulate Matter (see Appendix B).

1.2.5 NBS-SRM 1645, River Sediment (see Appendix C).

1.2.6 EPA Quality Control Samples - Trace Metals - WPI, or equivalent.

1.3 Preparation of Apparatus

All laboratory ware used for extraction, analysis and storage will be cleaned in the following manner (unless a variation which equals or exceeds the following methods has been approved by the evaluation team):

1.3.1 Wash in hot water using a laboratory detergent.

1.3.2 Rinse thoroughly three times with hot tap water to remove all of the detergent.

1.3.3 Immerse in 1:5 (v/v) nitric acid bath and allow to soak for a minimum of four hours.

1.3.4 Remove from the acid bath and rinse three times with deionized water in order to remove all of the acid.

1.3.5 Dry the glassware at 105°C.

1.3.6 Protect all cleaned laboratory ware from dust and fumes by appropriate means (e.g., cover with aluminum foil, put in plastic bag, store in closed containers, etc.)

1.4 Stock and Working Solution Preparations

1.4.1 Prepare 1:1 (v/v) HNO_3 used for extractions by diluting 500 ml concentrated HNO_3 with D.I. water to 1 liter.

1.4.2 Prepare 1:4 (v/v) HNO_3 used for preparation of standard solutions by diluting 250 ml conc. HNO_3 with D.I. water to 1 liter.

1.4.3 Prepare 1:5 (v/v) HNO_3 used for acid wash by diluting 200 ml of concentrated HNO_3 with D.I. water to 1 liter. Prepare appropriate volume necessary for a polyethylene acid wash tank. Analyze acid wash for lead on every working day and keep record of the daily lead values. Renew solution when a daily analysis shows lead content to be $\geq$ 5 ppm.

1.4.4 Prepare 1:100 (v/v) HNO_3 used for rinsing and for extract dilution by diluting 10 ml conc. HNO_3 with D.I. water to 1 liter.

1.4.5 Prepare 1000 µg/ml Pb standard stock solution by dissolving 1.5985 g of NBS-SRM 928, $Pb(NO_3)_2$, in 200 ml of D.I. water contained in a 1000-ml volumetric flask. Acidify with 250 ml conc. HNO_3 and dilute to volume with D.I. water. Record contents and date on label.

1.4.6 Prepare 100 µg/ml Pb standard solution by diluting 50 ml of 1000 µg/ml Pb standard stock solution to volume in a 500-ml volumetric flask with 1:4 HNO_3. This solution must be prepared fresh on each sample analysis day.

1.4.7 Calibration solution preparation:

Prepare fresh calibration standards of 0.5, 1.0, 2.0, 5.0, 10.0, and 15.0 µg/ml Pb, respectively, on an as needed basis by pipetting 0.5, 1.0, 2.0, 5.0, 10.0 and 15.0 mL, respectively, of the 100 µg/ml standard into 100-ml volumetric flasks and bringing to volume with 1:4 HNO_3.

1.5 Calibration of Equipment

1.5.1 Analytical Balance

Weigh a set of class S weights ranging from 1 to 100 g on the analytical balance(s) used during this study at the beginning of the study and repeat every 21 days. If any of the readings do not agree within ±0.2 mg with the stated value, have the balance recalibrated. Record the results of all balance checks in the balance log book. A calibration should also be performed whenever the analytical balance has been moved or has been subjected to rough handling or cleaning.

1.5.2 Atomic Absorption Spectrophotometer

a. Prepare a set of 6 calibration standards when atomic absorption analyses are performed, as described in Section 1.4.7.

b. With the 6 calibration standards ranging in concentration from 0.5 to 15 μg/ml, and a blank consisting of 1:4 (v/v) HNO_3 (see 1.4.2), calibrate the atomic absorption spectrophotometer using the procedures recommended by the manufacturer.

c. Plot on graph paper the absorbance data versus concentration and eye-estimate the linear portion of the calibration curve. For the linear region enter into a linear regression algorithm the absorbance versus concentration data to determine the working calibration graph to which subsequent sample absorbances are compared to yield concentration.

1.6 Sample Preparation and Extraction

Two basic types of samples will be collected and analyzed for lead during this study, namely soil samples and dust wipes (cotton swabs which have been used to wipe dust from the interior of houses located in the target area as specified in the House Dust Sampling Protocol).

1.6.1 Soil

The participating laboratory will receive soil sample aliquots consisting of approximately 60 g of soil which has been previously dried, screened, mixed and split. The samples will be contained in 27-ounce whirl-pak polyethylene containers. The soil samples will be prepared for extraction using the following procedures:

a. Hand-mix in its receiving container each soil sample received by tumbling the container 3 to 5 times.

b. Grind with mortar and pestle approximately 5 grams (Note) of sample, sieve through a 100-mesh sieve, and weigh into a tared beaker. Dry for one hour at 105°C, cool, and weigh to the nearest 0.1 mg to determine the exact sample weight. Store the remaining original sample in the original container in a locked room until written permission has been received from EPA stating that the samples can be discarded.

Note 1: After every use, mortar, pestle and sieve shall be cleaned as follows: Wipe off all soil with tissue paper. Rinse three times with D.I. water. Wipe dry with tissue paper. When not in use, mortar, pestle and sieve must be protected from dust and fumes as specified in 1.3.6.

Note 2: The sample size of 5 grams was chosen over 1 gram because the larger sample size will reduce errors due to sample inhomogeneity.

c. Cover the beaker containing the weighed sample with a watch glass and keep it in a secured area until the extraction is performed.

d. To each soil sample add 50 ml of 1:1 HNO_3 and re-cover the beaker with a watch glass.

e. Heat the sample at 95°C and gently boil for 2 hours. Stir sample occasionally with a glass rod. If during this step the volume goes below 25 ml, add more 1:1 HNO_3.

f. Allow the sample to cool, add 10 ml of 1:100 (v/v) HNO_3 and filter through Whatman No. 42 filter paper (or equivalent) into a 100-ml volumetric flask. Rinse the watch glass, glass rod, beaker and filter paper with two 10-ml portions of 1:100 HNO_3 and collect rinsings in the volumetric flask. Bring to volume with 1:100 HNO_3. The sample is now ready for analysis.

1.6.2 Dust Wipe Samples

It is anticipated that each dust wipe sample will consist of two individual swabs.

a. With teflon-coated tweezers, transfer the dust wipe sample swabs to a beaker for extraction. Dust wipe samples are not weighed as the analytical results are reported as total micrograms of lead per square centimeter wiped.

Note: Make sure that both swabs which make up one individual sample are placed in the same beaker.

b. Cover the beaker containing the dust wipe sample with a watch glass and keep it in a secured area until the extraction is performed.

c. To each sample consisting of 2 swabs, add 30 ml of 1:1 HNO_3 and cover the beaker with a watch glass.

d. Heat the sample at 95°C and gently boil for 2 hours. Stir sample occasionally with a glass rod. If during this step the volume goes below 15 ml, add more 1:1 HNO_3.

e. Allow the sample to cool, add 10 ml of 1:100 (v/v) HNO_3 and filter through Whatman No. 42 filter paper (or equivalent) into a 50-ml volumetric flask. Rinse the watch glass, glass rod, beaker and filter paper with two 10-ml portions of 1:100 HNO_3 and collect rinsings in the volumetric flask. Bring to volume with 1:100 HNO_3. The sample is now ready for analysis.

1.7 Atomic Absorption Analysis for Lead

1.7.1 Install lead hollow cathode lamp. Set wavelength of the monochromator at 283.3 nm. Set instrument operating conditions as recommended by the manufacturer. Background correction is required for these analyses.

1.7.2 At the beginning of each analysis set, but at least once per shift, calibrate the instrument as described in 1.5.2. Immediately following calibration, aspirate a known control sample such as the EPA QA Water Samples - WPI, independent of the calibration standards. Refer to Section 2.1.2 for details on use.

1.7.3 Aspirate sample extracts into flame. Take the mean of three integrations per sample (integration time = 5 to 7 seconds) and record this value (equilibrium absorbance) and the sample number on the strip chart tracing (or equivalent). Dilute 1:10 all samples, that exceed the highest working standard, with 1:4 HNO_3 so that their absorbances are within the linear working range. If the 1:10 dilution is not sufficient then dilute the original extract 1:100 with 1:4 HNO_3.

1.7.4 After each 10 samples aspirate one calibration standard of 10 µg/ml. If the standard result deviates by more than 5 percent from the value predicted by the calibration curve, recalibrate and repeat the previous 10 analyses.

1.7.5 After every 20 samples aspirate the known control sample as in 1.7.2.

1.8 Data Reduction and Reporting

1.8.1 Soil sample analytical results are to be expressed as micrograms of lead per gram of soil (dry weight) and are calculated using the following equation:

$$\mu g\ Pb/gm\ (dry\ weight) = ABC/D$$

where A = µg/ml of Pb in sample extract
B = Final volume of sample extract (100 ml)
C = Dilution factor (10 or 100), if necessary
D = Sample weight in grams (dry weight)

The minimum reportable level is 10 µg Pb/gm based on the lowest working standard of 0.5 µg Pb/ml. Samples whose extracts are below 0.5 µg Pb/ml but are above the limit of detection (approximately 0.2 µg Pb/ml) should be reported but the values should be bracketed ( ), indicating that the results were detected but are below the lowest working standard. Samples at or below the detection limit are reported as N.D. (Not Detected).

1.8.2 Results from dust wipe samples are to be expressed in micrograms of lead per square centimeter of area wiped and are calculated using the following equation:

$$\mu g\ Pb/cm^2 = ABC/D$$

where A = µg/ml of Pb in sample extract

B = Final volume of sample extract (50 ml)

C = Dilution factor (10 or 100), if necessary

D = 100 cm^2

The minimum reportable level is 0.25 µg Pb/cm^2 based on the lowest working standard of 0.5 µg Pb/ml. Samples whose extracts are below 0.5 µg Pb/ml but are above the limit of detection (approximately 0.2 µg Pb/ml) should be reported but the values should be bracketed ( ), indicating that the results were detected but are below the lowest working standard. Samples at or below the detection limit are reported as N.D. (Not Detected).

1.8.3 The results are reported for sets of 20 samples to the designated organizations on Sample Analysis Report Forms (Figures 1 and 2) together with all required QC data which are listed for each set of 20 samples on a separate form (Figures 3 and 4).

DALLAS LEAD SMELTER STUDY

Soil Sample Analysis Report

Report to: Date Reported:

Distribution:

Laboratory Name:

Date Sample Received	Field Sample ID	Lab Sample ID	Date Extraction Performed, Analyst Initials	Date Analysis Completed, Analyst Initials	Result µg Pb/gm Soil

Laboratory Supervisor

Figure 1

DALLAS LEAD SMELTER STUDY

Housedust Sample Analysis Report

Report to: Date Reported:

Distribution:

Laboratory Name:

Date Sample Received	Field Sample ID	Lab Sample ID	Date Extraction Performed, Analyst Initials	Date Analysis Completed, Analyst Initials	Result µg Pb/cm^2

Laboratory Supervisor

Figure 2

SOIL SAMPLE QUALITY ASSURANCE

Report to: ____________________ Date Reported: ____________________

Distribution: ____________________ QC Report # ____________________

____________________ Sample #'s: (Sequence as analyzed)

____________________ ____________________

____

Laboratory name: ____________________

Laboratory supervisor: ____________________ Analyst(s) initials: ________

1. Reagent blank: µg/ml ____________________
   (see 2.1.1)

2. Field blank: µg/ml ____________________
   (see 2.1.3)

   Sample # ____________________

3. Calibration control sample: µg/ml:
   (see 2.1.2)

   Found ____________________

   True ____________________

   Source: ____________________

   % recovery ____________________

4. Spike extract analysis: µg/ml

   Sample # ____________________
   (see 2.1.5)

   Sample extract result ____________________

   Spike extract result ____________________

   Spike added ____________________

   % recovery ____________________

Figure 3. (continued)

5. Residue re-extraction analysis: µg/gm (see 2.1.6)

Sample # ____________

Original extraction ____________

Residue extraction ____________

% originally extracted ____________

---

6. Total recoverable analysis: µg/gm (see 2.1.7)

Sample # ____________

Original result ____________

"Total" method result ____________

---

7. Laboratory control sample analysis: µg/gm (see 2.1.8)

Source: NBS SRM 1645, River Sediment

Found ____________

True ____________

% recovery ____________

---

8. Split extract analysis: µg/gm

Sample # ____________ (see 2.1.9)

Original extract result ____________

Split extract result ____________

---

9. Spiked sample analysis: µg/gm

Sample # ____________ (see 2.1.10)

Spiked sample result ____________

Unspiked sample result ____________

Spike added ____________

% recovery ____________

Figure 3

DUST WIPE SAMPLE QUALITY ASSURANCE

Report to: ____________________
Date Reported: ____________________

Distribution: ____________________
____
____

QC Report # ____________________

Sample #'s: (Sequence as analyzed)
____
____

Laboratory name: ____________________

Laboratory supervisor: ____________________ Analyst(s) initials: ________

---

1. Reagent blank: µg/ml ____________________
(see 2.2.1)

---

2. Field blank: µg/ml^2 ____________________
(see 2.2.3)

Sample # ____________________

---

3. Calibration control sample: µg/ml:
(see 2.2.2)

Source: ____________________

Found ____________________
True ____________________
% Recovery ____________________

---

4. Spike extract analysis: µg/ml
(see 2.2.4)
Sample # ____________________

Sample extract result ____________________
Spike extract result ____________________
Spike added ____________________
% Recovery ____________________

---

5. Laboratory control sample analysis: weight % Pb
(see 2.2.5)

Source: NBS SRM 1648, Urban Particulate

Found ____________________
True ____________________
% Recovery ____________________

---

6. Split extract analysis: µg/cm^2
Sample # ____________________
(see 2.2.6)

Original extract result ____________________
Split extract result ____________________

Figure 4

2.0 Quality Control Procedures

The following quality control analyses will be performed at the stated frequencies:

Soil:

a. one reagent blank (3 per shift day - at beginning, middle, and end or maximum 1 per 20 field samples)

b. one calibration control sample (one per 20)

c. Field and sample bank blanks (one per 40: field blank; one per 40: sample bank blank)

d. one split sample (blind)(one per 20 - other split sent to NEIC)

e. one spiked extract (one per 20)* where the parent sample is X_6

f. one reextraction of sample residue (one per 20)* where the parent sample is X_{12}

g. one "total recoverable" analysis (one per 10)* where the parent samples are X_9 for g_1 and X_{20} for g_2

h. one laboratory control sample (one per 20)*

i. one split extract analysis (one per 20)* where the parent sample is X_8

j. one spiked sample (one per 20)* where the parent sample is X_9

Dust Wipes:

a. one reagent blank (3 per shift day - at beginning, middle and end or maximum 1 per 20 field samples)

---

*The frequency of these analyses may be decreased during the study.

b. one calibration control sample (one per 20)

c. one field blank (one per 20)

d. one spiked extract (one per 20)* where the parent sample is X_{10}

e. one laboratory control sample (one per 20)*

f. one split extract (one per 20)* where the parent sample is X_8

In order to minimize bias the QC samples are intermixed with the field samples and the analysis sequence following calibration and analysis of the calibration control sample (b) will be randomly changed. The laboratory shall order the analysis sequence for every set of 20 samples as follows for soil (and similarly for dust), and then refer to a random number table to determine where to start the analysis sequence.

1. Perform calibration and calibration control samples analysis. After every tenth sample, reanalyze a calibration standard (see Section 1.7.4).

---

*The frequency of these analyses may be decreased during the study.

2. Order 20 field samples (X_1 to X_{20}) and corresponding QC samples as follows: X_1, X_2, X_3, a, X_4, X_5, X_6, e, X_7, X_8, X_9, g_1, X_{10}, X_{11}, X_{12}, f, X_{13}, X_{14}, X_{15}, h, X_{16}, X_{17}, X_{18}, i, X_{19}, X_{20}, g_2, j, . . . continue with b and next sequence
where: X_1 . . . X_{20} are samples 1-20 and
where: a, e, g, f, h, i, j, and b are the QC analyses specified above. [QC samples c and d are not included since they are to be treated as samples.]

3. Refer to a random number table to determine where to start the analysis sequence (X_1 or e or etc.).

4. For dust wipe analyses a similar type of sequence is to be used. X_1 . . . X_5, a, X_6 . . . X_{10}, d, X_{11} . . . X_{15}, e, X_{16} . . . X_{20}, f . . . continue with b and next sequence.

2.1 Soil Analysis Quality Control

2.1.1 Reagent Blank Analysis

a. A reagent blank will be processed and analyzed at the rate of three blanks per shift day (beginning, middle and end of shift) or a maximum of one per each set of 20 samples, on each day that soil extractions are performed, to establish the background lead level.

b. The reagent blank analysis will be performed as outlined in Section 1.6.1 for the soil samples except that no soil sample is added. Results are to be reported on the Soil Sample Quality Assurance Form. If the concentration of lead in the reagent blank exceeds 0.25 μg/ml (one-half the lowest calibration standard) then sources of contamination should be identified and eliminated, and EMSL-LV should be contacted immediately for assistance.

2.1.2 Calibration Control Sample

a. In order to verify the instrument calibration, a known control sample, such as the EPA Quality Assurance Water Samples - Trace Metals - WPI (prepared by EMSL-CIN), will be analyzed immediately following calibration and after every 20 samples (see Section 1.7.5). These standards will be provided by EMSL-LV in a concentrated form. The participating laboratory will dilute the concentrate to fall within the calibration range of the AAS for lead.

b. Calculate the percent recovery, using the certified lead concentration of the control sample, as diluted, and the analyzed concentration, according to the following equation:

$$\% \text{ recovery} = A/B \times 100$$

where: A = analyzed concentration of lead
B = certified concentration of lead (as diluted).

Results are to be reported on the Soil Sample Quality Assurance Form.

c. Maintain control charts on the percent recovery and establish through repetitive analyses the 99 and 95% confidence limits (control and warning).

d. If the percent recovery is outside of the acceptable range of 95 to 105% or outside the 99% confidence limits, whichever is less, then corrective action such as recalibration and preparation of fresh working and/or stock lead solutions must be taken. EMSL-LV will be available for assistance if required.

2.1.3 Field and Sample Bank Blank Analyses

For every 40 soil samples collected in the field one soil container filled with DI water will be sent to the Sample Bank and then forwarded to the participating laboratory. For every 40 soil samples processed through the sample bank, DI water will be similarly processed and then forwarded to the participating laboratory in a soil container. For each blank, the entire contents of the container will be transferred to a beaker, carefully evaporated to near dryness and then processed as specified in Section 1.6.1 for soil samples. Results are to be reported on the Soil Sample Quality Assurance Form.

2.1.4 Split Sample Analysis (Blind)

One in 20 soil samples sent to the Sample Bank will be split and forwarded as a blind duplicate to the participating laboratory.

2.1.5 Spiked Extract Analysis

For each 20 soil samples, one sample extract is spiked with lead nitrate to determine the effect of the extract matrix on the accuracy of the analysis procedure. The parent soil sample will be sample X_6.

a. The predetermined soil sample is extracted and analyzed as previously described (Section 1.6.1).

b. To an aliquot of this sample extract or its final dilution, if applicable, is added an amount of lead (as lead nitrate) approximately equal to the lead content determined for this extract or its final dilution, if applicable, but greater than 10 x the instrumental detection limit. This is best accomplished with 1000 ug/ml Pb standard stock solution using a calibrated micropipet such that the extract volume is not appreciably changed.

c. Directly analyze the spiked extract as described in Section 1.7.

d. Calculate the percent recovery of the spike using the following equation:

$$\% \text{ recovery} = (A-B)/C \times 100$$

where: A = analyzed total lead content of the spiked extract
B = analyzed lead content of the unspiked extract
C = lead content of spike.

Results are to be reported on the Soil Sample Quality Assurance Form. If the percent recovery is outside of the acceptable range of 90 to 110% then repeat the spiked extract analysis on two additional sample extracts from the same set of 20 samples. If the result from either of the additional spiked extracts is outside the acceptable range then use the method of additions for analysis of all 20 samples as described in EPA-600/4-79-020, March 1979 (Appendix D).

2.1.6 Reextraction of Sample Residue

a. For each 20 samples, one sample residue consisting of the filter paper and the undissolved soil portion will be extracted and analyzed according to the procedures set forth in Section 1.6.1. The parent sample will be X_{12}.

b. Calculate the percent of lead extracted by the first extraction based on the total of two extractions as follows:

$$\% \text{ extracted} = \frac{A}{A + B} \times 100$$

where: A = ug Pb/gm from first extraction

B = ug Pb/gm from second extraction (based on original sample weight).

c. Report results on the Soil Sample Quality Assurance Form.

2.1.7 "Total Recoverable" Analysis

For each 10 samples, a "total recoverable" analysis must be performed. The parent samples will be X_9 for g_1 and X_{20} for g_2. The extraction method chosen for this step has been demonstrated to give equivalent results for a variety of soil types when compared to known "total" methods. However, it is still necessary to closely monitor the extraction efficiency throughout this study. Once sufficient data have been gathered, reevaluation of the frequency of the "total recoverable" analysis may be considered. It is expected that the results from the "total recoverable" method detailed below will agree within 10% with those generated using the method detailed in Section 1.6.1. If after the analysis of 200 samples this agreement can be verified then the frequency of the "total recoverable" analyses might be lowered.

Note: The more rigorous $HF\text{-}HClO_4\text{-}HNO_3$ method which is widely used for total metals analysis in soil was considered but not chosen. This method is overly cumbersome, time-consuming and requires special equipment, and it was felt that any additional information that may be gained using the HF method would not warrant the additional effort.

DIGESTION PROCEDURE

Weigh and transfer to a 150 ml beaker a 5.0-gram portion of sample which has been prepared as discussed in Section 1.6.1. Add 25 ml of 1:1 HNO_3 and cover with a watch glass. Heat the sample at 95°C and reflux to near dryness. Allow the sample to cool, add 10 ml of conc. HNO_3 and again reflux to near dryness. After the second reflux step has been completed and the sample has cooled, add 5 ml of 1:1 HNO_3 and 3 ml of 30% hydrogen peroxide (H_2O_2). Return the beaker to the hot plate for warming to start the peroxide reaction. Care must be taken with the start of effervescence that losses do not occur or the reaction is not too vigorous. Heat until effervescence subsides and cool the beaker. Continue the addition of 30% H_2O_2 in 1-ml aliquots with warming until the effervescence is minimal or the general sample appearance is unchanged. Do not add more than a total of 10 ml 30% H_2O_2.

Add 5 ml of 1:1 HNO_3, return the covered beaker to the hot plate and reflux for an additional 10 min. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to

100 ml with 1:4 nitric acid. The sample is now ready for analysis according to Section 1.7.

Results are to be reported in ug Pb/gm on the Soil Sample Quality Assurance Form. If the "total recoverable" analysis results differ by more than 10% from the results obtained according to 1.6 and 1.7, EMSL-LV should be contacted immediately for assistance.

2.1.8 Laboratory Control Sample Analysis.

a. One NBS-SRM 1645, River Sediment, sample will be analyzed for each 20 field soil samples, as an independent certified control sample.

b. The 5-gram samples are to be prepared and analyzed as detailed in Appendix C and in Section 1.6.1.b through f (except for the grinding and sieving) and 1.7.

c. Report on the Soil Sample Quality Assurance Form the results and the percent recovery of NBS certified concentration as calculated below:

$$\% \text{ recovery} = B/A \times 100$$

where: A = NBS SRM certified lead concentration

B = analyzed lead concentration

d. Maintain control charts on the percent recovery and establish through repetitive analyses the 99 and 95% confidence limits (control and warning).

e. If the percent recovery is outside of the acceptable range of 90 to 110%, or outside the 99% confidence limits, whichever is less, the analysis will be repeated. If the percent recovery is outside the acceptable range with the repeat sample, a thorough check of the extraction and analysis procedures should be conducted before proceeding with additional field sample analyses. EMSL-LV should be contacted immediately for assistance.

2.1.9 Split Extract Analysis

Intralaboratory precision for the analysis only will be estimated from the results of two replicate aliquots (split extracts) from every twentieth sample:

a. For each 20 soil sample extracts, split the extract from sample X_8 (see page 22) into two portions. Analyze the first portion as extract X_8, the second portion as i following X_{18}.

b. Report the results of the split extract analysis on the Soil Sample Quality Assurance Form.

A precision of 3% is expected. If the precision is outside of the 5% range, EMSL-LV must be notified immediately.

2.1.10 Spiked Sample Analysis

In order to monitor potential lead losses during extraction, a spiked sample analysis will be performed on a duplicate portion of one sample for each 20 field samples. Once sufficient data have been gathered, the frequency of the spiked sample analysis may be reconsidered.

a. For each 20 samples, a duplicate portion of sample X_9 is spiked with 700 μg Pb, as lead nitrate solution, per gram of sample, and processed as specified in 1.6 and 1.7. EPA may request a change of the amount of lead added during the course of the study.

b. Calculate the percent recovery of the spike as follows:

$$\% \text{ recovery} = \frac{A - B}{C} \times 100$$

where: A = ug Pb/gm determined in spiked sample

B = ug Pb/gm determined in unspiked sample

C = ug Pb/gm added.

Results are to be reported on the Soil Sample Quality Assurance Form.

c. Maintain control charts on the percent recovery and establish through repetitive analyses the 99 and 95% confidence limits (control and warning).

d. No limits can be set at this time due to unknown sample homogeneity. However, if the percent recovery is outside of the 99% control limits, repeat the spiked sample analysis on a second sample from the same set of 20 samples. If this sample recovery is also outside of the 99% control limits, contact EMSL-LV immediately for assistance.

2.2 Dust Wipe Analysis Quality Control.

2.2.1 Reagent Blank Analysis.

a. A reagent blank will be processed and analyzed at the rate of three blanks per shift day (beginning, middle and end of shift) or a maximum of one per each set of 20 samples, on each day that dust wipe extractions are performed, to establish the background lead level.

b. The reagent blank analysis will be performed as outlined in Section 1.6.2 for the dust wipe samples except that no swabs are added. Results are to be reported on the Dust Wipe Sample Quality Assurance Form. If the concentration of lead in the reagent blank exceeds 0.25 ug/ml (one-half the lowest calibration standard), then sources of contamination should be identified and eliminated, and EMSL-LV should be contacted immediately for assistance.

2.2.2 Calibration Control Sample.

a. In order to verify the instrument calibration, a known control sample, such as the EPA Quality Assurance Water Samples-Trace

Metals-WPI (prepared by EMSL-CIN), will be analyzed immediately following calibration and after every 20 samples (see Section 1.7.5). These standards will be provided by EMSL-LV in a concentrated form. The participating laboratory will dilute the concentrates to fall within the calibration range of the AAS for lead.

b. Calculate the percent recovery, using the certified lead concentration of the control sample (taking into consideration the dilution) and the analyzed concentration, according to the following equation:

$$\% \text{ recovery} = A/B \times 100$$

where: A = analyzed concentration of lead
B = certified concentration of lead (as diluted).

c. Maintain control charts on the percent recovery and establish through repetitive analyses the 99 and 95% confidence limits (control and warning).

Results are to be reported on the Dust Wipe Sample Quality Assurance Form. If the percent recovery is outside of the acceptable range of 95 to 105% or outside the 99% confidence limits, whichever is less, then corrective action, such as recalibration and preparation of fresh working and/or stock lead solutions must be taken. EMSL-LV will be available for assistance if required.

2.2.3 Field Blank Analysis.

For every 20 dust wipe samples collected in the field, one field blank (consisting of two swabs) will be delivered to the laboratory. The field blank sample will be processed and analyzed as specified in Section 1.6.2. Results are to be reported on the Dust Wipe Sample Quality Assurance Form. The field blanks will be clearly identified as such on the labels and accompanying forms.

2.2.4 Spiked Extract Analysis.

For each 20 field dust samples, one sample extract is spiked as follows with lead nitrate to determine the effect of the extract matrix on the accuracy of the analysis procedure. The parent dust wipe sample will be sample X_{10}.

a. The predetermined dust wipe sample is extracted and analyzed as previously described (Section 1.3).

b. To an aliquot of this sample extract or its final dilution, if applicable, is added an amount of lead (as lead nitrate) approximately equal to the lead content determined for this extract or its final dilution, if applicable, but greater than 10 x the instrumental detection limit. This is best accomplished with 1000 ug/ml Pb standard stock solution using a calibrated micropipet such that the extract volume is not appreciably changed.

c. Directly analyze the spiked extract as described in Section 1.7.

d. Calculate the percent recovery of the spike using the following equation:

$$\% \text{ recovery} = (A-B)/C \times 100$$

where: A = analyzed total lead content of the spiked extract

B = analyzed lead content of the unspiked extract

C = lead content of spike.

Results are to be reported on the Dust Wipe Sample Quality Assurance Form. If the percent recovery is outside of the acceptable range of 90-110%, then repeat the spiked extract analysis on two additional sample extracts from the same set of 20 samples. If the result from either of the additional spiked extracts is outside the acceptable range, then use the method of additions for analysis of all 20 samples as described in EPA-600/4-79-020, March 1979 (Appendix D).

2.2.5 Laboratory Control Sample Analysis.

For each 20 field dust wipe samples, one laboratory control sample (NBS SRM 1648, Urban Particulate Matter) will be analyzed. The laboratory control sample is prepared in the laboratory as follows:

a. Dry the material as specified in Appendix B. Weigh 0.1 gram (to the nearest 0.1 mg) of NBS SRM 1648, Urban Particulate Matter, into a tared beaker and then carefully add two freshly unwrapped dust swabs.

b. Analyze the control sample as described in Section 1.6.2.

c. Report on the Dust Wipe Quality Assurance Form the results as % Pb of the Urban Particulate Matter, and calculate the percent recovery based on the NBS certified concentration as follows:

$$\% \text{ recovery} = B/A \times 100$$

where: A = NBS SRM certified concentration
B = analyzed lead concentration.

d. Maintain control charts on the percent recovery and establish through repetitive analyses the 99 and 95% confidence limits (control and warning).

e. If the percent recovery is outside of the acceptable range of 95 to 105%, or outside the 99% confidence limits, whichever is less, prepare a fresh control sample and repeat the extraction and analysis procedures. If on the repeat sample the percent recovery is outside of the acceptable range of 95 to 105%, or the 99% confidence limits, whichever is less, a thorough check of the extraction and analysis procedures should be conducted before proceeding with additional field samples. EMSL-LV should be contacted immediately for assistance.

2.2.6 Split Extract Analysis

Both the overall measurement precision and the intralaboratory precision will be estimated from the split extract analysis.

a. For each 20 dust wipe sample extracts, split the from sample X_8 (see page 23) into three portions.

b. Send one fraction to the referee laboratory, NEIC, with all proper identification and chain-of-custody information.

c. Analyze the second portion as extract X_8 and the third portion as f following X_{20}.

d. Report the results of the split extract analysis on the Dust Sample Quality Assurance Form.

U.S. Department of Commerce
Elliot L. Richardson,
Secretary

National Bureau of Standards
Ernest Ambler, Acting Director

Appendix A

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 928
Lead Nitrate
(Clinical Standard)

This Standard Reference Material is certified for use as an assay standard for lead. It is intended primarily for use in the calibration and standardization of procedures employed in clinical analysis and for the routine critical evaluation of daily working standards used in these procedures.

Assay . . . . 100.00 ± 0.03 percent

The assay shown is based on the determination of lead in the material as received; drying being unnecessary. Lead is precipitated as the chromate using a slight excess of potassium dichromate (SRM 136c). The lead chromate is removed by filtration and the excess chromate ion determined spectrophotometrically. Details of this method are reported elsewhere [1]. The molecular weight of lead nitrate employed in the calculation is 331.219. This value is based on a mass-spectrometrically determined value of 207.209 for the atomic weight of lead in this sample. The uncertainty shown represents two standard deviations of a single measurement based on 16 determinations with allowances for known sources of possible error.

A semi-quantitative survey for trace metals by emission spectroscopy indicated the following: silver, 2 µg/g; chromium, 3 µg/g; nickel, 3 µg/g. No other metals were detected.

The lead nitrate used for this Standard Reference Material was obtained from the J. T. Baker Chemical Co., Phillipsburg, New Jersey. Chemical analyses were performed by T. J. Murphy and J. W. Gramlich. Spectroscopic analyses were performed by C. S. Annell and D. Golightly of the United States Geological Survey.

The overall direction and coordination of technical measurements leading to certification were under the chairmanship of I. L. Barnes.

The technical and support aspects concerning preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D.C. 20234
February 27, 1976
Revised May 19, 1976

J. Paul Cali, Chief
Office of Standard Reference Materials

This material was examined for compliance with the specifications for reagent grade lead nitrate as given in Reagent Chemicals, 5th edition, published by the American Chemical Society. The material met or exceeded the requirements in every respect.

This Standard Reference Material is intended for "in vitro" diagnostic use only.

This material is intended for use as a standard for lead determination in clinical chemistry.

Because of the instability of non-acidified aqueous lead solutions at the working levels it is recommended [2] that three levels of concentration be used.

(a) A stock standard solution containing 50 mmol/l is prepared by dissolving 1.6561 g of SRM 928 in ion-free water. If the solution is cloudy or a precipitate forms, add a few drops of ammonium hydroxide. Mix, dilute to 100 ml in a calibrated volumetric flask and transfer immediately to a previously acid-washed, water-rinsed, dry polyethylene bottle. This solution is stable for six months. [2].

The above directions are directly quoted from the reference given. Users are cautioned that carbonate-free ammonium hydroxide not in excess of 0.2 ml should be added. Otherwise, insoluble basic lead salts will form. However, experience at NBS indicates that this material will easily dissolve *without cloudiness* when high-purity water is used.

(b) An intermediate solution containing 500 μmol/l is prepared by a 1:100 dilution of the above stock solution. This solution may be stored in a capped polyethylene bottle at room temperature for one month [2].

(c) Working standard solutions of 0.5, 1.0, 2.5, and 5.0 μmoles/l should be prepared each time an analysis is performed [2].

Note: Dilute aqueous lead standards remain stable for less than three hours. Lead is readily adsorbed on the surfaces of glass and plastic containers and this reaction is accelerated by exposure to light, particularly ultraviolet light [3]. It is recommended that very dilute aqueous lead solutions be prepared in a darkened room and protected from light [4].

This Standard Reference Material should be stored in the tightly-closed original bottle under normal laboratory conditions. Tests show the material to be dry as received and not to adsorb appreciable water when exposed to a 90 percent relative humidity atmosphere for 5 days.

The solutions of SRM 928 are stable as described above. At time of use these solutions should be clear and display no turbidity.

References:

[1] E. J. Catanzaro, T. J. Murphy, W. R. Shields and E. L. Garner, J. Res. NBS 72A, 261-267 (1968).

[2] L. Kopito and H. Shwachman, Measurement of lead in blood, urine, and hair by atomic absorption spectroscopy, Standard Methods of Clinical Chemistry, Vol. 7, G. R. Cooper, editor-in-chief, pp. 151-162, Academic Press, Inc., New York, N.Y. 1972.

[3] L. Kopito and H. Shwachman, J. Lab. Clin. Med. 70, 326-332 (1967).

[4] Fundamentals of Clinical Chemistry, N. W. Tietz, editor, pp. 852-857, W. B. Saunders Co., Philadelphia, Pa. (1970).

The date of issuance and certification of this Standard Reference Material was June 15, 1975.

APPENDIX B

S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards
Certificate of Analysis

Standard Reference Material 1648

Urban Particulate Matter

This Standard Reference Material is intended for use in the calibration of methods used in the chemical analysis of atmospheric particulate matter and materials with similar matrices. The material is atmospheric particulate matter collected in an urban location.

The certified values are based on measurements of 6 to 30 samples by each of the analytical techniques indicated. The estimated uncertainties include those due to sample variation, possible methodology differences, and errors of measurement (see Preparation and Analysis). The certified values are based on a sample size of at least 100 mg of the dried material. The material should be dried at 105 °C for 8 hours before use.

Element	μg/g	Element	Weight %
Arsenic[e,c]	115 ± 10	Iron[a,b,c,e]	3.91 ± 0.10
Cadmium[a,b,c,d]	75 ± 7	Lead[a,b,d]	0.655 ± .008
Chromium[b,c]	403 ± 12		
Copper[a,b,c]	609 ± 27		
Nickel[a,b,d]	82 ± 3		
Zinc[a,b,c,d]	4760 ± 140		
Uranium[b]	5.5 ± 0.1		

[a] Atomic Absorption Spectrophotometry
[b] Isotope Dilution Mass Spectrometry
[c] Neutron Activation Analysis
[d] Polarography
[e] Spectrophotometry

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J. K. Taylor.

The technical and support aspects involved in preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

Washington, D.C. 20234
November 16, 1978

J. Paul Cali, Chief
Office of Standard Reference Materials

Preparation and Analysis

This SRM was prepared from urban particulate matter collected in the St. Louis, Missouri, area in a baghouse especially designed for this purpose. The material was collected over a period in excess of 12 months and, therefore, is a time-integrated sample. While not represented to be typical of the area in which it was collected, it is believed to typify the analytical problems of atmospheric samples obtained from industrialized urban areas.

The material was removed from the filter bags by a specially designed vacuum cleaner and combined into a single lot. This product was screened through a fine-mesh sieve to remove most of the fibers and other extraneous material from the bags. The sieved material was then thoroughly mixed in a V-blender, bottled, and sequentially numbered.

Randomly selected bottles were used for the analytical measurements. Each analyst examined at least 6 bottles, some of them measuring replicate samples from each bottle. No correlation was found between measured values and the bottling sequence. Also, the results of measurements of samples from different bottles were not significantly different than the measurements of replicate samples from single bottles. Accordingly, it is believed that all bottles of this SRM have the same composition.

The analytical methods employed were those in regular use at NBS for certification of Standard Reference Materials, except as noted in the following paragraphs. Measurements and calibrations were made to reduce random and systematic errors to no more than one percent, relative. The uncertainties of the certified values listed in the table include those associated both with measurement and material variability. They represent the 95 percent tolerance limits for an individual sub-sample, i.e., 95 percent of the sub-samples from a single unit of this SRM would be expected to have a composition within the indicated range of values 95 percent of the time.

The following values have not been certified because either they were not based on results of a reference method, or were not determined by two or more independent methods. They are included for information only. All values are in units of $\mu g/g$ of sample, unless otherwise indicated.

Aluminum	(3.3 wt. %)	Lanthanum	(42)
Antimony	(45)	Magnesium	(0.8 wt. %)
Barium	(737)	Manganese	(860)
Bromine	(500)	Potassium	(1.0 wt. %)
Cerium	(55)	Samarium	(4.4)
Cesium	(3)	Scandium	(7)
Chlorine	(0.45 wt. %)	Selenium	(24)
Cobalt	(18)	Silver	(6)
Europium	(0.8)	Sodium	(0.40 wt. %)
Hafnium	(4.4)	Thorium	(7.4)
Indium	(1.0)	Titanium	(0.40 wt. %)
Iodine	(20)	Tungsten	(4.8)
Vanadium	(130)		

The values listed below are based on measurements made in a single laboratory, and are given for information only. While no reason exists to suspect systematic bias in these numbers, no attempt was made to evaluate such bias attributable to either the method or the laboratory. The method used for each set of measurements is also listed. The uncertainties indicated are two times the standard deviation of the mean.

Nitrogen (NO_3)	(1.07% ± 0.06)
Nitrogen (NH_4)	(2.01% ± .08)
Sulfate	(15.42% ± .14)
SiO_2	(26.8% ± .38)
Freon Soluble	(1.19% ± .47)

The above values were determined by the methods indicated below:

Nitrate – Extraction with water and measurement by ASTM Method D992.

Ammonia – NaOH addition followed by steam distillation and titration.

Sulfate – Extraction with water and measurement by ASTM D516.

SiO_2 – Solution and measurement by ASTM Method E350.

Freon Soluble – Extraction with Freon 113, using the method described in "Standard Methods in Examination of Water and Waste Water," 14th Edition, p. 518, American Public Health Association, Washington, D.C.

J. W. Matwey supervised the collection of the material as well as sieving and bottling. The following members of the staff of the NBS Center for Analytical Chemistry performed the certification measurements: R. W. Burke; E. R. Deardorff; B. I. Diamondstone; L. P. Dunstan; M. S. Epstein; M. Gallorini; E. L. Garner; J. W. Gramlich; R. R. Greenberg; L. A. Machlan; E. J. Maienthal; and T. J. Murphy.

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1645

River Sediment

This Standard Reference Material is intended for use in the calibration of methods used in the analysis of river sediment and materials with similar matrices. The material has been freeze dried and is now essentially free from moisture. The certified values given below are based on measurements made on a dried sample of at least 100 mg for the trace elements and for a 1-g sample for iron and chromium.

The values are based on the results of 6 to 30 determinations by the analytical techniques indicated. The estimated uncertainties include those due to sample variation, possible method differences, and errors of measurement (see Preparation and Analysis).

Element	µg/g	Element	µg/g
Cadmium[c,d]	10.2 ± 1.5	Thorium[b]	1.62 ± 0.22
Copper[b,c]	109 ± 19	Uranium[b]	1.11 ± .05
Lead[b,d]	714 ± 28	Vanadium[a,c]	23.5 ± 6.9
Manganese[b,c]	785 ± 97	Zinc[a,d]	1720 ± 169
Mercury[a,c]	1.1 ± 0.5		Weight %
Nickel[b,d]	45.8 ± 2.9	Chromium[b,c]	2.96 ± 0.28
Thallium[b]	1.44 ± 0.07	Iron[a,c]	11.3 ± 1.2

[a] Atomic Absorption Spectrometry
[b] Isotope Dilution Mass Spectrometry
[c] Neutron Activation Analysis
[d] Polarography

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J. K. Taylor.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

Washington, D.C. 20234
November 16, 1978

J. Paul Cali, Chief
Office of Standard Reference Materials

Instructions for Use

The material, as received, is essentially free from moisture. In case of exposure to moisture, it should be dried without heat to a constant weight before using. Recommended procedures for drying are: (1) drying for 24 hours using a cold trap at or below −50 °C and a pressure not greater than 30 Pa (0.2 mm Hg); (2) drying in a desiccator over P_2O_5 or $Mg(ClO_4)_2$. When not in use, the material should be kept in a tightly sealed bottle and stored in a cool, dark place.

Material of this kind is intrinsically heterogeneous. Consequently, the analyst should endeavor to minimize any segregation by thoroughly mixing the contents of the bottle by shaking and rolling before each use. In addition, when taking a portion for analysis, the analyst should strive to remove as representative a sample as possible.

Preparation and Analysis

This SRM was prepared from material dredged from the bottom of the Indiana Harbor Canal near Gary, Indiana. This material was screened to remove foreign objects, freeze dried, and sieved to pass a No. 80 (180μm) screen. This material was thoroughly mixed in a V-blender, bottled, and sequentially numbered. The material has been radiation-sterilized to minimize alteration from biological activity.

Randomly selected bottles were used for the analytical measurements. Each analyst examined at least 6 bottles, some of them measuring replicate samples from each bottle. No correlation was found between measured values and the bottling sequence. The results of measurements on samples from different bottles did not appear to differ significantly from sub-samples within the bottles. Accordingly, it is believed that all bottles of this SRM have substantially the same composition. The analytical methods employed were those in regular use at NBS for certification of Standard Reference Materials, except as noted below. Measurements and calibrations were made to reduce random and systematic errors to no more than one percent, relative. The uncertainties of the certified values listed in the table include those associated with both measurement and material variability. They represent the 95 percent tolerance limits for an individual sub-sample, i.e., 95 percent of the sub-samples from a unit of this SRM would be expected to have a composition within the indicated range of values 95 percent of the time.

The following values have not been certified because either they are not based on results of a reference method, or were not determined by two or more independent methods. They are included for information only.

All values are in units of μg/g of sample, unless otherwise indicated.

Antimony	(51)	Potassium	(1.2 wt. %)
Arsenic	(66)	Scandium	(2)
Cobalt	(8)	Sodium	(0.55 wt. %)
Lanthanum	(9)		

The values listed below are based on measurements made in one laboratory, and are given for information only. While no reason exists to suspect systematic bias in these numbers, no attempt was made to evaluate such bias attributable to either the method or the laboratory. The method used for each set of measurements is also listed. The uncertainties indicated are two times the standard deviation of the mean.

Kjeldahl Nitrogen	(0.0797% ± 0.0048)
Total Phosphorus	(.051% ± .0014)
Loss on Ignition (800 °C)	(10.72% ± .28)
Oil and Grease (Freon)	(1.71% ± .26)
Chemical Oxygen Demand (Dichromate)	(149,400 mg/kg ± 9,000)

The methods used are:

Total Phosphorus - ASTM Method E-350.

Chemical Oxygen Demand (Dichromate) - Standard Methods for the Examination of Water and Waste Water, 14th Edition (1975), Section 508, page 550.

Oil and Grease (Freon 113 Extraction) - ibid., Section 502, page 518.

The following values are not certified, but are given to describe the matrix of the material: SiO_2 - 51%; MgO - 4%; Al_2O_3 - 4%; CaO - 4%.

H. L. Rook supervised collection, freeze drying, and homogenization of the SRM. The following members of the staff of the NBS Center for Analytical Chemistry performed the certification measurements: T. J. Brady; E. R. Deardoff; L. P. Dunstan; M. S. Epstein; R. Filby; M. Gallorini; E. L. Garner; T. E. Gills; J. W. Gramlich; R. R. Greenberg; S. H. Harrison; G. J. Lutz; L. A. Machlan; E. J. Maienthal; T. C. Rains; H. L. Rook; T. A. Rush; and W. P. Schmidt.

The development work, preceeding the certification of the SRM, was supported by the Environmental Protection Agency under an Interagency Agreement.

DN-126-82
EAD
7-14-88

(See Note 8) Cool and add 15 ml of carbon disulfide in small portions and with swirling. Dilute to 1 liter with chloroform. The solution can be used for several months if stored in a brown bottle in a refrigerator.

NOTE 8: An acceptable grade of pyrrolidine may be obtained from the Aldrich Chemical Co., 940 West St. Paul Ave., Milwaukee, WI 53233 (414-273-3850).

7.7.2 Ammonium hydroxide, 2N: Dilute 3 ml conc. NH_4OH to 100 ml with deionized distilled water.

7.7.3 Bromphenol blue indicator (1 g/liter): Dissolve 0.1 g bromphenol blue in 100 ml of 50 percent ethanol or isopropanol.

7.7.4 HCl,2.5% v/v: Dilute 2 ml redistilled HCl to 40 ml with deionized distilled water.

8. Preparation of Standards and Calibration

8.1 Stock standard solution are prepared from high purity metals, oxides or nonhygroscopic reagent grade salts using deionized distilled water and redistilled nitric or hydrochloric acids. (See individual analysis sheets for specific instruction.) Sulfuric or phosphoric acids should be avoided as they produce an adverse effect on many elements. The stock solutions are prepared at concentrations of 1000 mg of the metal per liter. Commercially available standard solutions may also

8.2 Calibration standards are prepared by diluting the stock metal solution at the time of analysis. For best results, calibration standards should be prepared fresh each time an analysis is to be made and discarded after use. Prepare a blank and at least four calibration standards in graduated amounts in the appropriate range. The calibration standards should be prepared using the same type of acid or combination of acids and at the same concentration as will result in the samples following processing. As filtered water samples are preserved with 1:1 redistilled HNO (3 ml per liter), calibration standards for these analyses should be similarly prepared with HNO. Beginning with the blank and working toward the highest standard, aspirate the solutions and record the readings. Repeat the operation with both the calibration standards and the samples a sufficient number of times to secure a reliable average reading for each solution. Calibration standards for furnace procedures should be prepared as described on the individual sheets for that metal.

8.3 Where the sample matrix is so complex that viscosity, surface tension and components cannot be accurately matched with standards, the method of standard addition must be used. This technique relies on the addition of small, known amounts of the analysis element to portions of the sample--the absorbance difference between those and the original solution giving the slope of the calibration curve. The method of standard addition is described in greater detail in (8.5).

8.4 For those instruments which do not read out directly in concentration, a calibration curve is prepared to cover the appropriate concentration range. Usually, this means the preparation of standards which produce an absorption of 0 to 80 percent. The correct method is to convert the percent absorption readings to absorbance and plot that value against concentration. The following relationship is used to convert absorption values to absorbance:

$$\text{absorbance} = \log(100/\%\,T) = 2 - \log \%\,T$$
$$\text{where } \%\,T = 100 - \%\text{ absorption}$$

As the curves are frequently nonlinear, especially at high absorption values, the number of standards should be increased in that portion of the curve.

8.5 Method of Standard Additions: In this method, equal volumes of sample are added to a deionized distilled water blank and to three standards containing different known amounts of the test element. The volume of the blank and the standards must be the same. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Fig. 1.

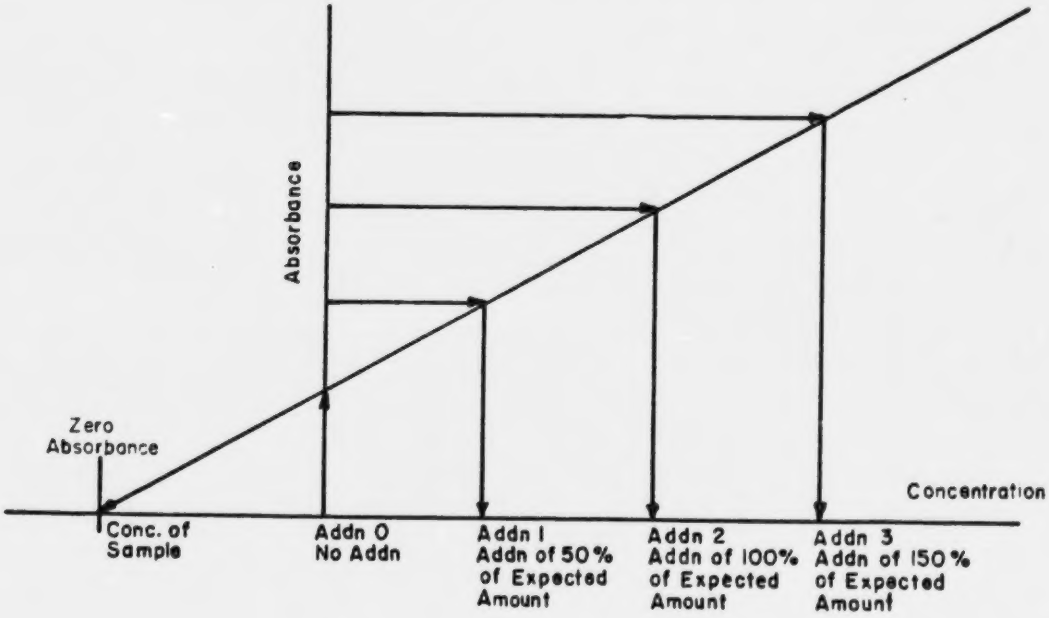


FIGURE 1. STANDARD ADDITION PLOT

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The method of standard additions can be very useful, however, for the results to be valid the following limitations must be taken into consideration:

a) the absorbance plot of sample and standards must be linear over the concentration range of concern. For best results the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%) caution should be exercised.

b) the effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes and the standard addition should respond in a similar manner as the analyte.

c) the determination must be free of spectral interference and corrected for non-specific background interference.

9. General Procedure for Analysis by Atomic Absorption

9.1 Direct Aspiration: Differences between the various makes and models of satisfactory atomic absorption spectrophotometers prevent the formulation of detailed instructions applicable to every instrument. The analyst should follow the manufacturer's operating instructions for his particular instrument. In general, after choosing the proper hollow cathode lamp for the analysis, the lamp should be allowed to warm up for a minimum of 15 minutes unless operated in a double beam mode. During this period, align the instrument, position the monochromator at the correct wavelength, select the proper monochromator slit width, and adjust the hollow cathode current according to the manufactuerer's recommendation. Subsequently, light the flame and regulate the flow of fuel and oxidant, adjust the burner and nebulizer flow rate for maximum percent absorption and stability, and balance the photometer. Run a series of standards of the element under analysis and construct a calibration curve by plotting the concentrations of the standards against the absorbance. For those instruments which read directly in concentration set the curve corrector to read out the proper concentration. Aspirate the samples and determine the concentrations either directly or from the calibration curve. Standards must be run each time a sample or series of samples are run.

9.1.1 Calculation - Direct determination of liquid samples: Read the metal value in mg/l from the calibration curve or directly from the readout system of the instrument.

9.1.1.1 If dilution of sample was required:

$$\text{mg/l metal in sample} = A\left(\frac{C + B}{C}\right)$$

where:

A = mg/l of metal indiluted aliquot from calibration curve
B = ml of deionized distilled water used for dilution
C = ml of sample aliquot

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9.1.2 For samples containing particulates:

$$\text{mg/l metal in sample} = A\left(\frac{V}{C}\right)$$

where:

A = mg/1 of metal in processed sample from calibration curve
V = final volume of the processed sample in ml
C = ml of sample aliquot processed

9.1.3 For solid samples: report all concentrations as mg/kg dry weight

9.1.3.1 Dry sample:

$$\text{mg metal/kg sample} = \frac{A \times V}{D}$$

where:

A = mg/1 of metal in processed sample from calibration curve
V = final volume of the processed sample in ml
D = weight of dry sample in grams

9.1.3.2 Wet sample:

$$\text{mg metal/kg sample} = \frac{A \times V}{W \times P}$$

where:

A = mg/1 of metal in processed sample from calibration curve
V = final volume of the processed sample in ml
W = weight of wet sample in grams
P = % solids

9.2 Special Extraction Procedure: When the concentration of the metal is not sufficiently high to determine directly, or when considerable dissolved solids are present in the sample, certain metals may be chelated and extracted with organic solvents. Ammonium pyrrolidine dithiocarbamate (APDC) (see footnote) in methyl isobutyl ketone (MIBK) is widely used for this purpose and is particularly useful for zinc, cadmium, iron, manganese, copper, silver, lead and chromium^{+6}. Tri-valent chromium does not react with APDC unless it has first been converted to the hexavalent form [Atomic Absorption Newsletter *6*, p 128 (1967)]. This procedure is described under method 218.3.

The name ammonium pyrrolidine dithiocarbamate (APDC) is somewhat ambiguous and should more properly be called ammonium, 1-pyrollidine carbodithioate (APCD), CAS Registry No. 5108–96–3.

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APPENDIX D

ATTACHMENT 4

PROPOSED SURVEY DESIGN FOR INVESTIGATING THE EFFECT OF TRAFFIC AND SMELTER LEAD EMISSIONS IN DALLAS, TEXAS

RTI/1864/36 -01 W
June 18, 1982

FINAL DRAFT

PROPOSED SURVEY DESIGN FOR INVESTIGATING THE EFFECT OF TRAFFIC LEAD EMISSIONS IN DALLAS, TEXAS

by

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I. INTRODUCTION

A. Overview and Purpose

During the fall of 1981, the City of Dallas conducted voluntary blood-lead screening programs in which approximately 12,000 individuals participated. Analysis of the data suggests that a source of lead exists in the vicinity of the intersection of Singleton Boulevard and Westmoreland Avenue. Two potential major sources were suggested by reviewers, emission from automobiles traveling along the two streets and the secondary lead smelter located near the intersection. Because the effects of both emissions sources are present in this area, it is difficult to isolate individual effects. Hence, it is proposed that a study be conducted with emphasis one distinguishing the relative contribution from each of these two sources. This proposal offers a scientific design that will overcome the major shortcoming of the previous screening program; that is, the self selected sample, the confounding of effects from the smelter and traffic as emission sources, and the limited amount of environmental data for preschool children with a range of blood lead values.

B. Study Areas

One study area will comprise approximately a circle of radius one mile centered at the intersections of Singleton Boulevard and Westmorland Avenue where one smelter is located. A second study area will include a geographic area that approximately coincides with a circle with a radius of one mile centered at the intersection of McGowan Street and Sargent Road, the location of a second smelter. A third study area to be used as a comparison or reference area will consist of a separate geographic area that approximately coincides with a circle of radius one half mile centered at another intersection purposefully chosen to be similar to Singleton and Westmorland with regard to daily traffic patterns and volume and demographic characteristics. Several areas along with traffic, demographic, and socioeconomic information is presented to assist in selecting a satisfactory reference area.

C. Study Population

The study populations consist of preschool children (0-5 years) residing in the study areas. Questionnaire information and blood specimens will be collected from each child selected in the study and from three environmental media, soil, paint and settled dust within and about the residences. Soil samples will also be selected for nonresidential play areas in the sample blocks. Data will also be collected on characteristics of the household and housing unit.

D. Population Parameters

For the children residing in these areas, several population parameters are of interest. These are the mean and variance of the blood lead level, mean and variance of free erythrocyte protoporphyrin (FEP), the proportion of preschool children with greater than 29 μg/dℓ lead in blood, proportion of preschool children with greater than 49 μg/dℓ FEP in blood and the proportion of preschool children with both greater than 29 μg/dℓ lead and greater than 49 μg/dℓ FEP. For the other three media, the means, percentiles, and variance of the lead concentration are of interest. If threshold levels are defined for these media, the proportions based on the thresholds can also be estimated. CDC feels that the mean blood lead level and proportion of preschool children with greater than 29 μg/dℓ lead are the most important parameters.

Estimates of these parameters may be used as surrogates to estimate the relative contributions of the two suspected sources of emissions as well as other sources identified in the questionnaires or from other intelligence. The study areas will be partitioned into groups (strata). Each group or stratum will be classified as High, Moderate or Low for each source of emissions based on distance. Table 1 summarizes the strata classifications for the study areas. The parameter estimates may be tested for differences among strata individually or simultaneously using multivariate techniques. Differences of other subpopulations can also be tested to the extent these are adequately present in the sample.

Table 1. Classification of Strata by Source and Level of Potential Exposure*

Study Area	Stratum	Smelter Potential Exposure	Traffic Potential Exposure
One**	11	High	High
	12	High	Low
	13	Medium	High
	14	Medium	Low
Two	21	High	High
	22	High	Low
	23	Medium	High
	24	Medium	Low
Three	31	Low	High
(Reference)	32	Low	Low

*Based on work performed by RTI.

**One option includes only the first four strata, the other option includes all six strata.

II. SAMPLE SIZE DETERMINATION

To distinguish the relative contribution of the smelter and auto emissions to the total lead exposure of persons residing near the study site, it is first necessary to estimate appropriate sample sizes for comparing parameter estimates. The blood and environmental data from the screening program was used to obtain estimates of the coefficients of variation (CV), where the CV = σ/μ, and is estimated by the sample standard deviation divided by the mean. Recall that such estimates are tenuous because of data collection methods (volunteer screening program) however, they should provide useful information for design purposes. Table 2 summarizes the blood lead concentration and FEP data that resulted in the screening program. Table 3 summarizes the environmental data collected from approximately 100 residences. Table 4 summarizes the screening program data for the two most important parameters and groups of interest. Table 5 presents the sample sizes required to detect differences of the magnitude estimated in the previous study between the 0-.5 mile circle and the .5 to 1.0 mile "donut". These sample sizes were calculated assuming simple random sampling and not taking into account the small populations in the study areas. Using these assumptions, a sample of 300 appears to be sufficient to detect the above difference between the proportions of preschool children with greater than 29 μg/dℓ bloodlead. Table 6 presents the recommended sample sizes after the preliminary estimate is adjusted for the small populations in the study area. Table 6 also includes the estimated number of sample housing units to be screened to result in the required number of preschool children.

Table 2. Summaries of Blood Data from Dallas Screening Program*

Measurement (μg/dℓ)	Number Screened	Mean	Variance	Coefficient of Variation (Percent)
Blood Lead	11,089	12.7	35.6	47
FEP	7,316	24.1	270	68

*

Table 3. Summary of Lead Concentrations in Environmental Specimens from Dallas Screening Program*

Media	Sample Size	Mean	Total Variance	Coefficient of Variation (Percent)	Variance Components	
					Among Residences	Within Residence
Paint (PPM)	168	2.19	23.7	220	16.7	7.0
Soil (PPM)	211	1,760	$10.0\text{x}10^{6}$	179	$7.0\text{x}10^{5}$	$9.30\text{x}10^{6}$
Dust Wipes (μg/cm^2)	225	5.00	177	266	68	109

---

*Specimens were taken only from residences in which persons with greater than 29 μg/dℓ blood lead were located, as identified inthe Dallas Health Department screening program conducted in the Fall of 1981.

Table 4. Summary of Selected Screening Program Data for Preschool Children*

Study Site	Number Screened	Sample Mean Blood-Lead (μg/dℓ)	Sample Variance Blood-Lead	Proportion >29 μg/dℓ	Estimated Population Count (1980 Census)	Estimated Percent Participating In Screening Program
Northwest (Area One) Distance (miles)						
0.0 - 0.5	237	22.3	59.3	.135	731	32
0.5 - 1.0	171	17.4	37.8	.047	823	21
1.0 - 2.0	285	15.2	39.3	.035	††	††
0.5 - 2.0	456	16.0	38.7	.039	††	††
Southeast (Area Two) Distance (miles)						
0.0 - 0.5	40	17.6	55.5	.100	210	19
0.5 - 1.0	101	15.9	32.7	.020	245	13
1.0 - 2.0	445	15.6	22.9	.009	††	††
Comparison Area** Distance (miles)						
> 2.0	374	14.0	27.8	.013	††	††
All Preschool Children†	1,811	16.4	41.8	.040	††	††

*Source: Calculated from Screening Program Data Conducted by the City of Dallas, Texas in the Fall of 1981.

**Based on all preschool children living greater than two miles from both smelter sites.

†Includes 158 preschool children not geographically coded.

††Not Calculated.

Table 5. Minimum Sample Sizes for Each Area to Detect Differences Among Selected Population Parameters ($\alpha = .05$, $\beta = .10$ and Infinite Population)**

Parameter	Study Area	Sample Size to Detect** Difference Between 0-.5 Area and .5-1.0 Area, Number of Preschoolers for Each Area
Mean of Blood-lead	Northwest	53
	Southeast	397
Proportion of Blood-Lead >29 μg/dℓ	Northwest	272
	Southeast	223

α is the probability of a Type I error; that is, concluding the parameters are unequal in the two areas when in fact they are equal.

β is the probability of a Type II error; that is concluding the parameters are equal when in fact they are not.

**Sample Size = $\frac{\text{Variance of Difference} * ({}^{z}\alpha + {}^{z}\beta)^2}{\text{Difference}}$ and differences being referred to are of the magnitude indicated by the screening program and presented in Table 4 where $z_\alpha = 1.96$ and $z_\beta = 1.645$.

Table 6. Recommended Sample Sizes

Study Area	Unadjusted Sample Size (n_0) Number of Preschoolers	Estimated Number of Preschool Children in the Population (N)	Estimated Number of Occupied HUs in the Population	Adjusted Sample Size (n_1)*	Estimated** Number of Sample HUs
Northwest					
0.0 - 0.5	300	731	1350	213	711
0.5 - 1.0	300	823	1389	220	706
Southeast					
0.0 - 0.5	300	210	408	124	346
0.5 - 1.0	300	745	1793	214	751
Reference area (preliminary, pending selection of specific reference area	300			200-300	

*$n_1 = n_0/(1 + n_0/N)$

**Based on a 75 percent response rate, the average number of preschool children per housing unit (HY) and the 1980 occupancy rate.

III. SURVEY DESIGNS

Survey designs for three study areas are presented. Because the details for constructing the sampling frame, stratifying the study areas, and selecting the participants are similar in each study area, the details will be presented only for the first.

A. Study Area One

1. Sampling Frame and Stratification

The study area is defined as the aggregate of all blocks (as defined by the U.S. Bureau of the Census) that approximate a circle of radius one mile and centered at the intersection of Singleton Boulevard and Westmoreland Avenue (see Figure 1). The blocks will be the primary or first stage sampling units (PSUs). The study area will be partitioned into four disjoint areas using (1) vehicular traffic volume data provided by the city of Dallas for the streets and (2) distance from the intersection. Figures 1 illustrates the study area and the distance strata.

2. Sample Selection

The 1980 Census data at the block level will be used to estimate the number of preschool children and housing units (HUs) in each block. Approximately forty blocks will be selected from the study area with probability proportional to their estimated size (estimated number of preschoolers). Table 6 above presents the estimated number of preschool children in the study area along with the estimated number of HUs to be selected in the sample.

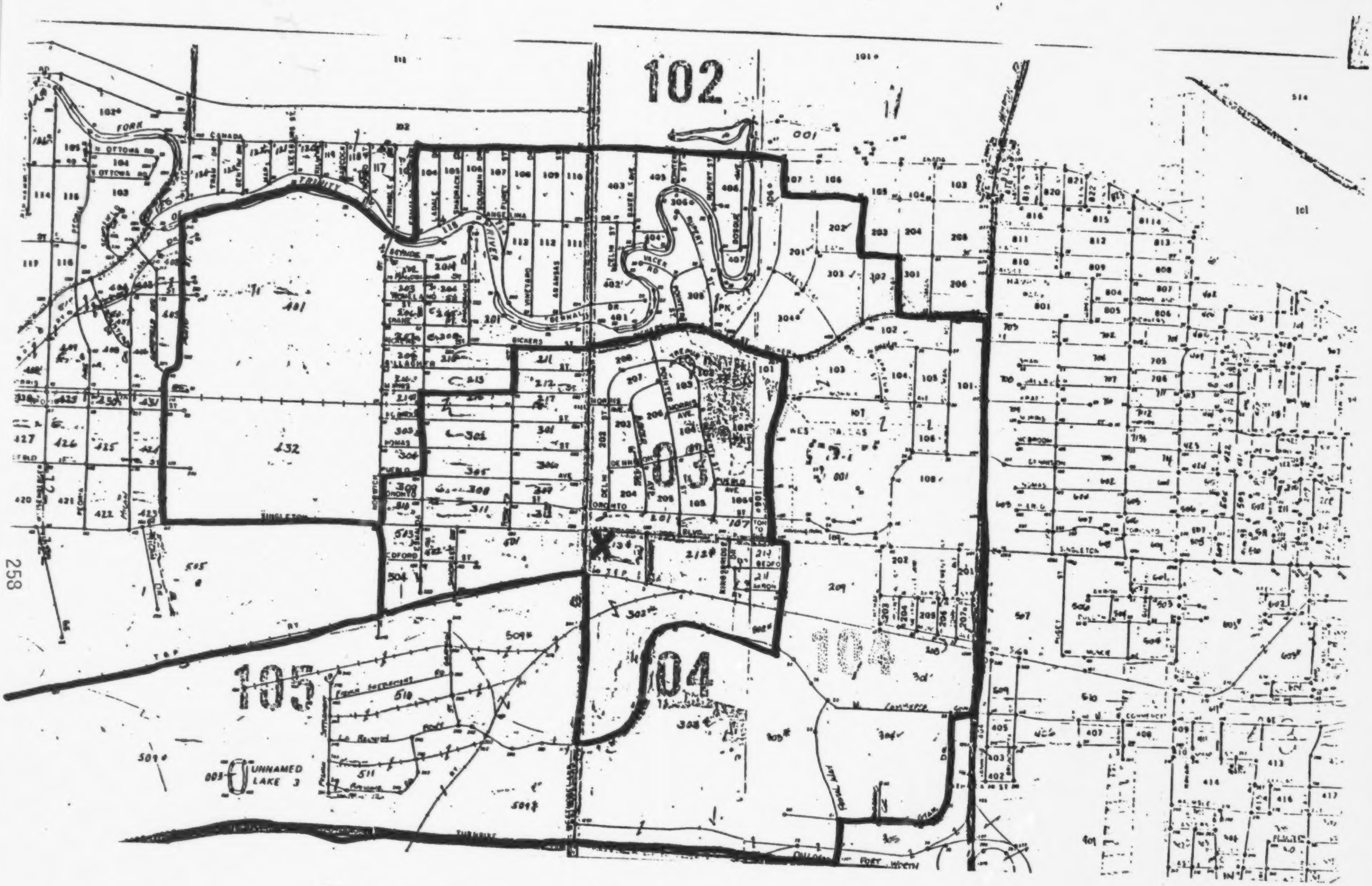
102
103
104
105
X
TRINITY
RIVER
FORK
UNNAMED LAKE 3
SINGLETON
TURNPIKE

Figure 1. Study Area One (Circular Option) With Distance Strata and Location of Smelter (X) Indicated

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A noncompact systematic sample of HUs will be selected from each PSU. The number of HUs selected in each PSU will be approximately equal to minimize the difference among sampling weights. To identify the sample HUs, an RTI employee will visit each selected PSU and sketch the locations and compile a list of all HUs in the block. The list is generated using a rigorously defined protocol and allows for later identification of the sample HUs. Figure 2 constitutes an example of a sketch provided to field personnel. Figure 3 depicts the same sketch after listing is completed. Figure 4 gives the list of housing units denoted on the sketch.

3. Data Collection

City of Dallas employees will contact the persons in the selected HUs to screen for preschool children, obtain cooperation, administer questionnaires, and collect environmental data. All preschool children in the selected HUs will be in the sample. Some incentive such or some service to the residence (free preschooler health exam) should be considered to aid in attaining a sufficient response rate (minimum of 75 percent). Repeated callbacks (number yet to be determined) are recommended to achieve the desired response rate. Environmental specimens such as soil, paint and dust will only be collected from the sample HUs where preschool children reside and from public play areas in selected blocks. Ideally, the sample locations for the environmental data, especially soil sampling, should be randomly selected. If not random, at least a protocol must be used that is clearly written and defines rigorously the exact sample locations to assure compariability of the measurements among HUs. This will help reduce the amount of variability in the data due to use of different sampling personnel. Also, with

LIST UNIT SKETCH

SEGMENT ID 12241-02-1-1 PLACE FAIRMONT, N.Y.

PREPARED BY Bill Watkins DATE 9/12/76

REVIEWED BY ______ DATE ______

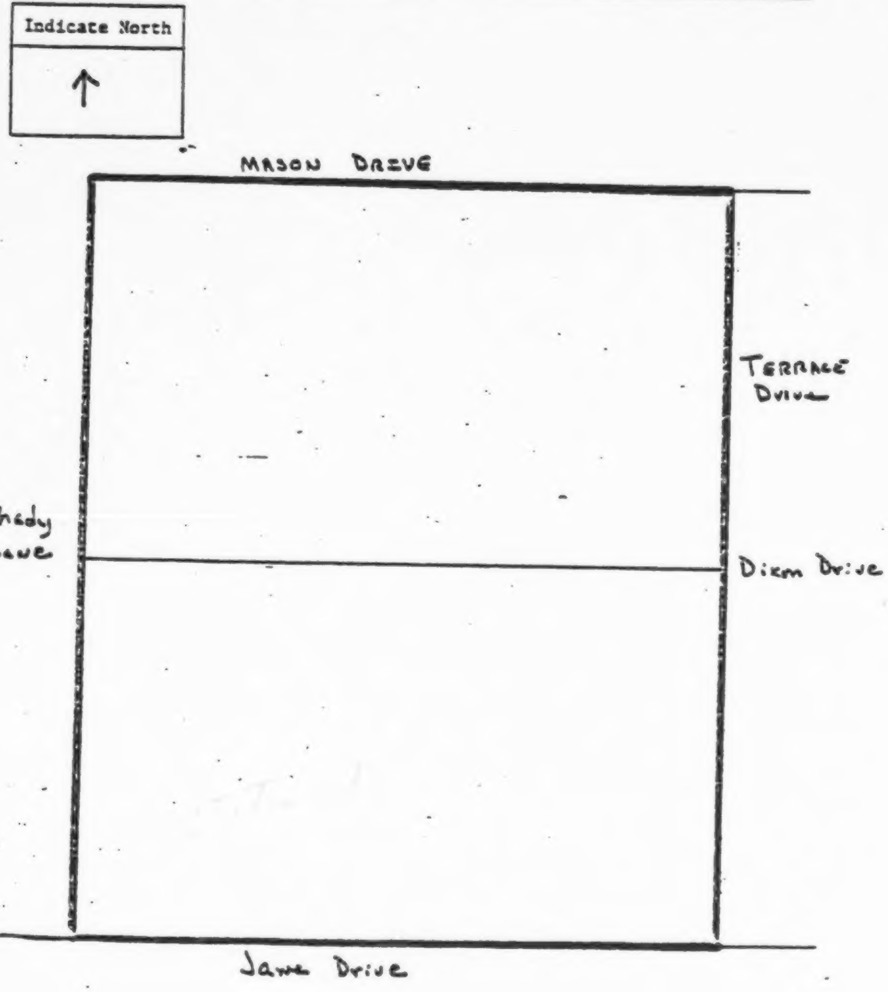
Indicate North
↑
MASON DRIVE
TERRACE Drive
Shady Lane
Dixon Drive
Jane Drive

Figure 2. Example of a List Unit Sketch Before Listing Performed

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LIST UNIT SKETCH

SEGMENT ID 12241-02-1-1 PLACE FAIRMONT, N.Y.

PREPARED BY Bill Watkins DATE 9/12/76

REVIEWED BY ______ DATE ______

Indicate North

↑

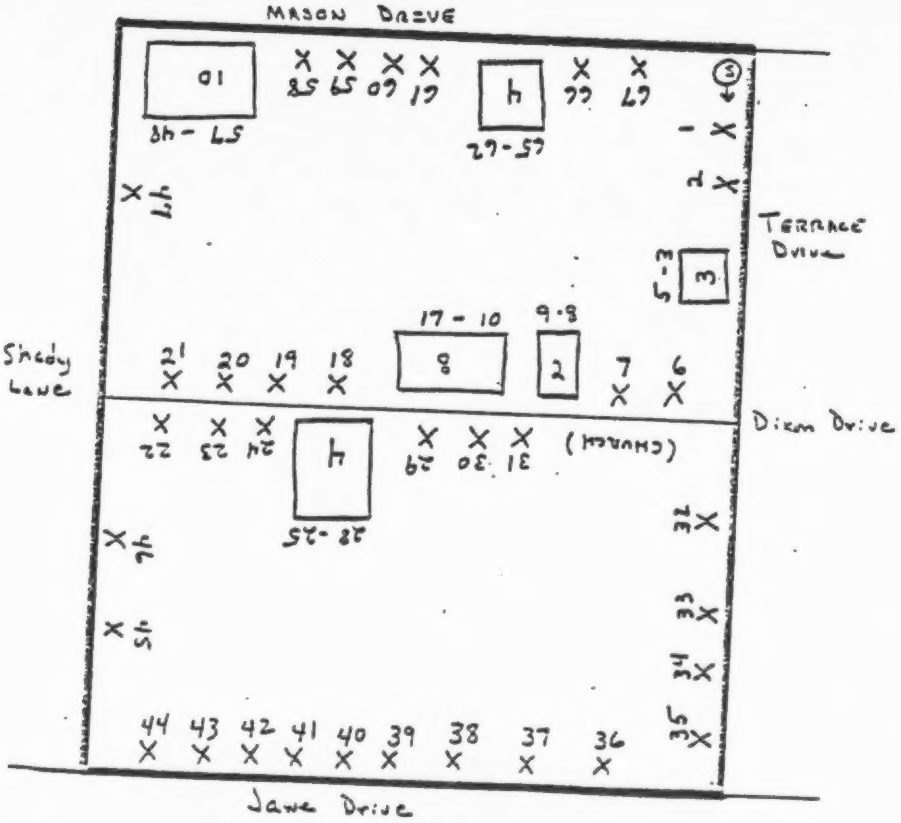
MASON DRIVE
10
57-48
58
59
60
61
4
62-65
66
67
1
2
47
TERRACE Drive
3
5-3
17-10
9-8
8
2
Shady Lane
21
20
19
18
7
6
Diem Drive
22
23
24
4
28-25
29
30
31
(CHURCH)
32
46
45
33
34
35
44
43
42
41
40
39
38
37
36
Jane Drive

Figure 3. Example of a List Unit Sketch after Listing Completed

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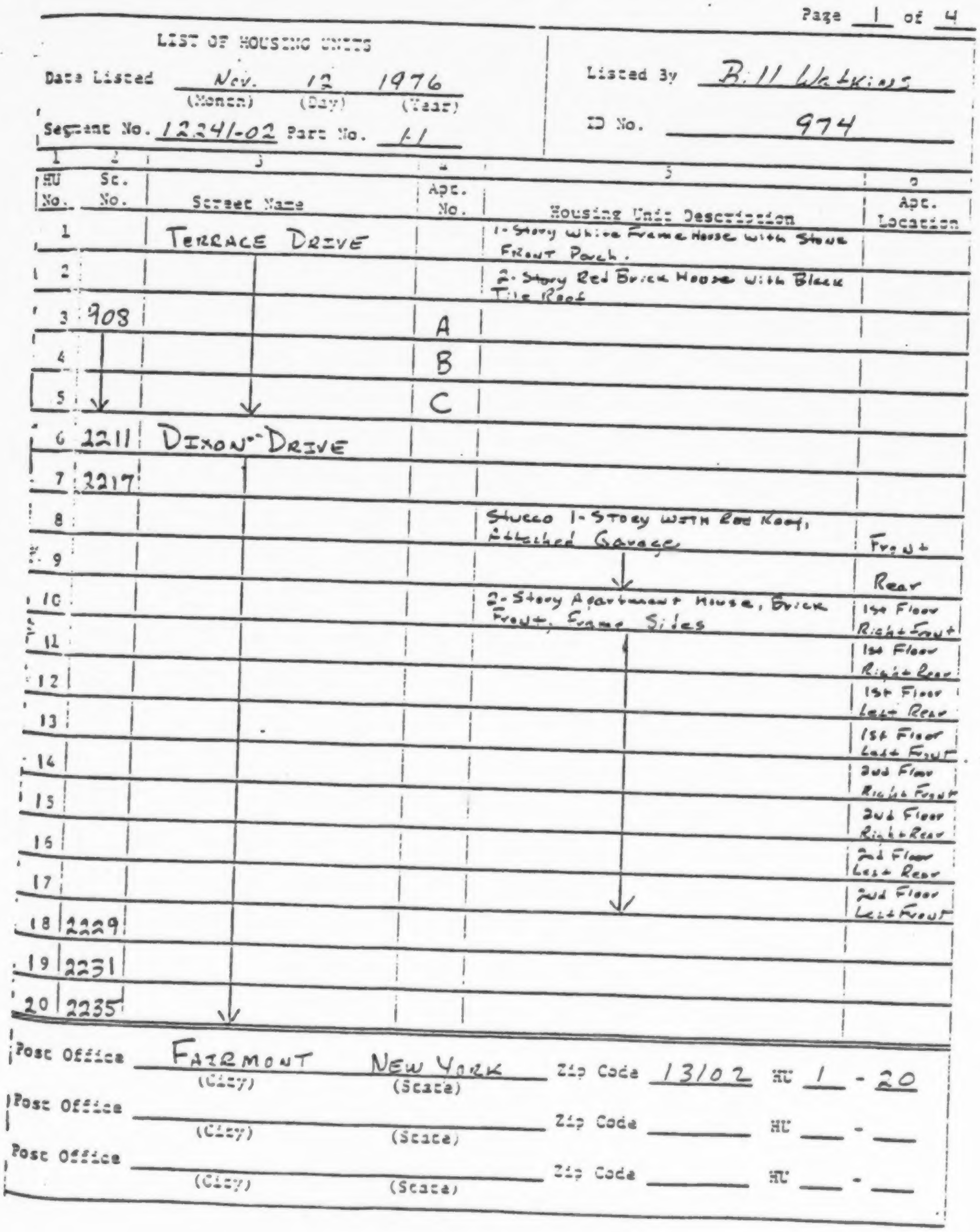

Page 1 of 4

LIST OF HOUSING UNITS

Date Listed Nov. 12 1976
(Month) (Day) (Year)

Segment No. 12241-02 Part No. 11

Listed By Bill Watkins

ID No. 974

1 HU No.	2 St. No.	3 Street Name	4 Apt. No.	5 Housing Unit Description	6 Apt. Location
1		Terrace Drive		1-Story White Frame House with Stone Front Porch.	
2				2-Story Red Brick House with Black Tile Roof	
3	908		A		
4			B		
5			C		
6	2211	Dixon Drive			
7	2217				
8				Stucco 1-Story with Red Roof, Attached Garage	Front
9					Rear
10				2-Story Apartment House, Brick Front, Frame Sides	1st Floor Right Front
11					1st Floor Right Rear
12					1st Floor Left Rear
13					1st Floor Left Front
14					2nd Floor Right Front
15					2nd Floor Right Rear
16					2nd Floor Left Rear
17					2nd Floor Left Front
18	2229				
19	2231				
20	2235				

Post Office Fairmont New York Zip Code 13102 HU 1 - 20
(City) (State)

Post Office ______ Zip Code ______ HU ___ - ___
(City) (State)

Post Office ______ Zip Code ______ HU ___ - ___
(City) (State)

Figure 4. List of Housing Units Contained in List Unit

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regard to soil sampling, the field staff should not be permitted to exclude certain locations in any attempt to obtain "representative" specimens (substitution of soil sampling points should follow a specific pattern and only be used if sampling is not feasible - pavement for example).

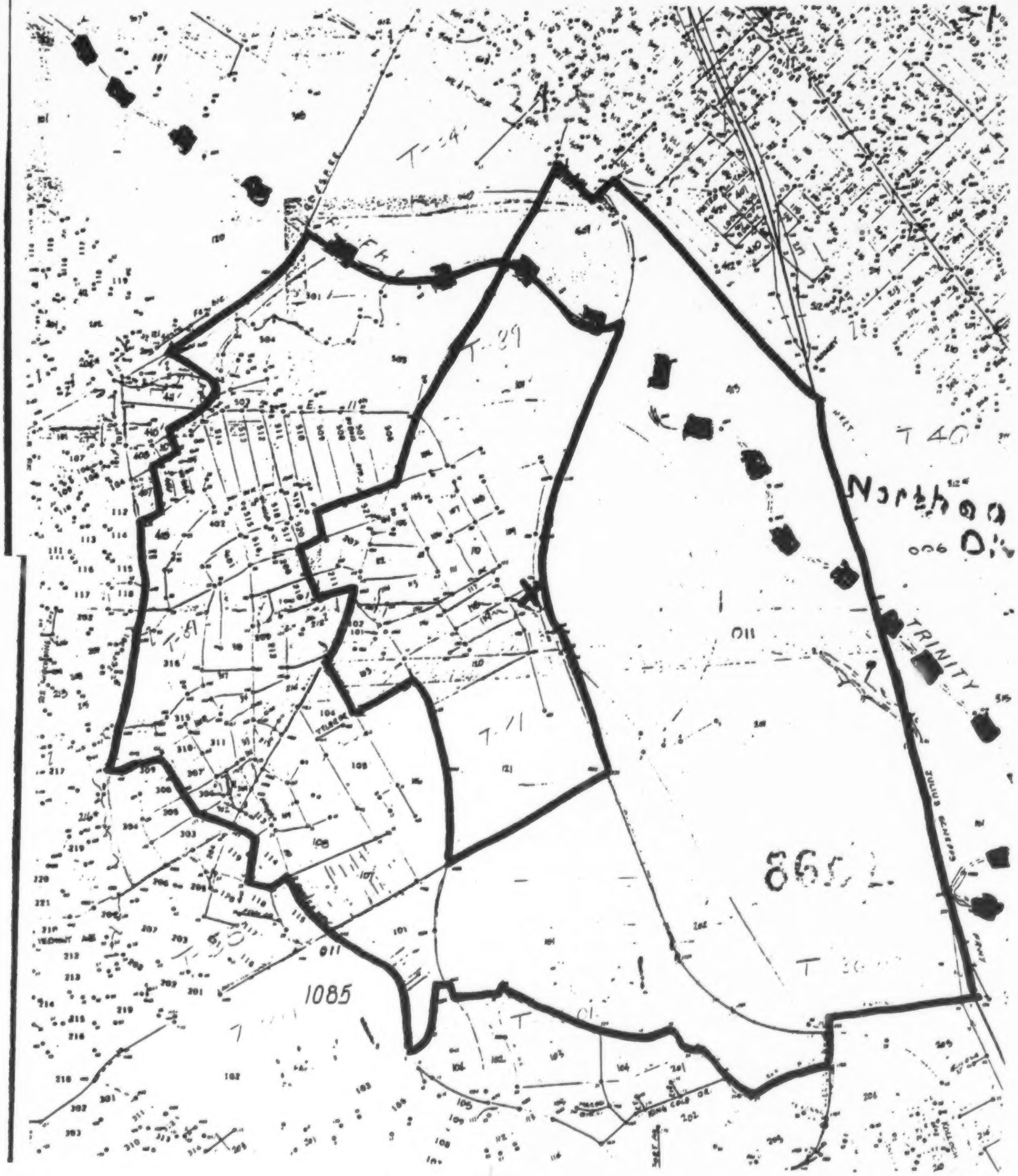
B. Study Area Two

Study area two is all blocks approximating a circle of radius one mile centered at the intersection of McGowan Street and Sargent Road., the location of the second smelter. Figure 5 illustrates the second study area and its strata. Table 6 above presents the estimated number of preschool children in the area along with the estimated number of HUs to be selected in the sample.

As in study area one, study area two will be stratified by proximity of the blocks to high traffic volume streets and proximity to a smelter. This will result in the four strata in the area described in Table 1 (page 3). The sample selection procedure and data collection guidelines are the same as in area one.

C. Study Area Three

In order to facilitate the effective selection comparison sites for this study, RTI compiled a set of descriptive statistics from the 1980 Census of Population (see Table 7). The locations of the candidate comparison areas are illustrated in Figures 6-8. These statistics are based on aggregate block level summaries and correspond roughly to one mile circular areas around the point sources in the case of study areas and on Census Track level summaries around the intersection of roads with available traffic volume data in the case of the comparison sites. These statistics only roughly correspond to the one mile areas because of the rectangular shape of some Census Tracts and the irregularity of shape for others. More detailed investigation for the most



T-41
T-39
T-40
TRINITY
1085

Figure 5. Study Area Two With Distance Strata and Smelter Location Indicated (X)

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Table 7. Summary of Descriptive Statistics for Study Areas and (Aggregate Block Level Data) at Census Tract Level for Comparison Sites*

	Study Areas		Dallas Reference Area**	Candidate Comparison Areas					
	One (RSR Corp.)	Two (Dixie Metals)		1	2	3	4	5	6
Total Population	10,237	6,997	20,063	28,122	16,544	24,924	21,333	23,882	27,899
Percent									
White	4.0	4.2	56.2	29.1	1.5	46.9	48.9	7.9	14.6
Black	88.3	87.5	33.3	67.8	98.1	39.6	43.4	87.4	84.0
Other	7.7	8.3	10.5	3.1	.4	13.5	7.7	4.7	1.3
Population 5 years or less of age									
Number	1554	955	2257	2968	1759	2733	2322	2408	2560
Percent of Total	15.2	13.6	11.25	10.55	10.60	10.96	10.88	10.08	9.18
Total Occupied Housing Units	2739	2201	6585	8904	4178	8754	7873	6536	8005
Total Persons Per Occupied Housing Unit	3.7	3.18	3.05	3.16	3.97	2.85	2.71	3.65	3.49
5 years or less Per Occupied Housing Unit	.57	.43	.34	.33	.42	.28	.29	.37	.32
24-Hour Traffic Volume Counts***									
N.S. Primary[1]/	17364	17151	11683	10913	6924	11573	9364	7586	6326
Secondary		6512							
E.W. Primary	11133		11806	10943	8161	22624	14454	14096	15,748
Secondary	65530[2]/								

---

[1]/Primary roadways are roadway bisecting the center of the study area.; secondary roadways are defined as those cutting across only part of the study area.

[2]/Traffice Volume of Dallas-Fort Worth Turnpike.

*Source: 1980 Census of Population and Housing. Summary Tape File B.

**

***Source: Automatic Traffice Recoder Data, Traffice County Operation, City of Dallas, Streets and Estimation Services, 1980.

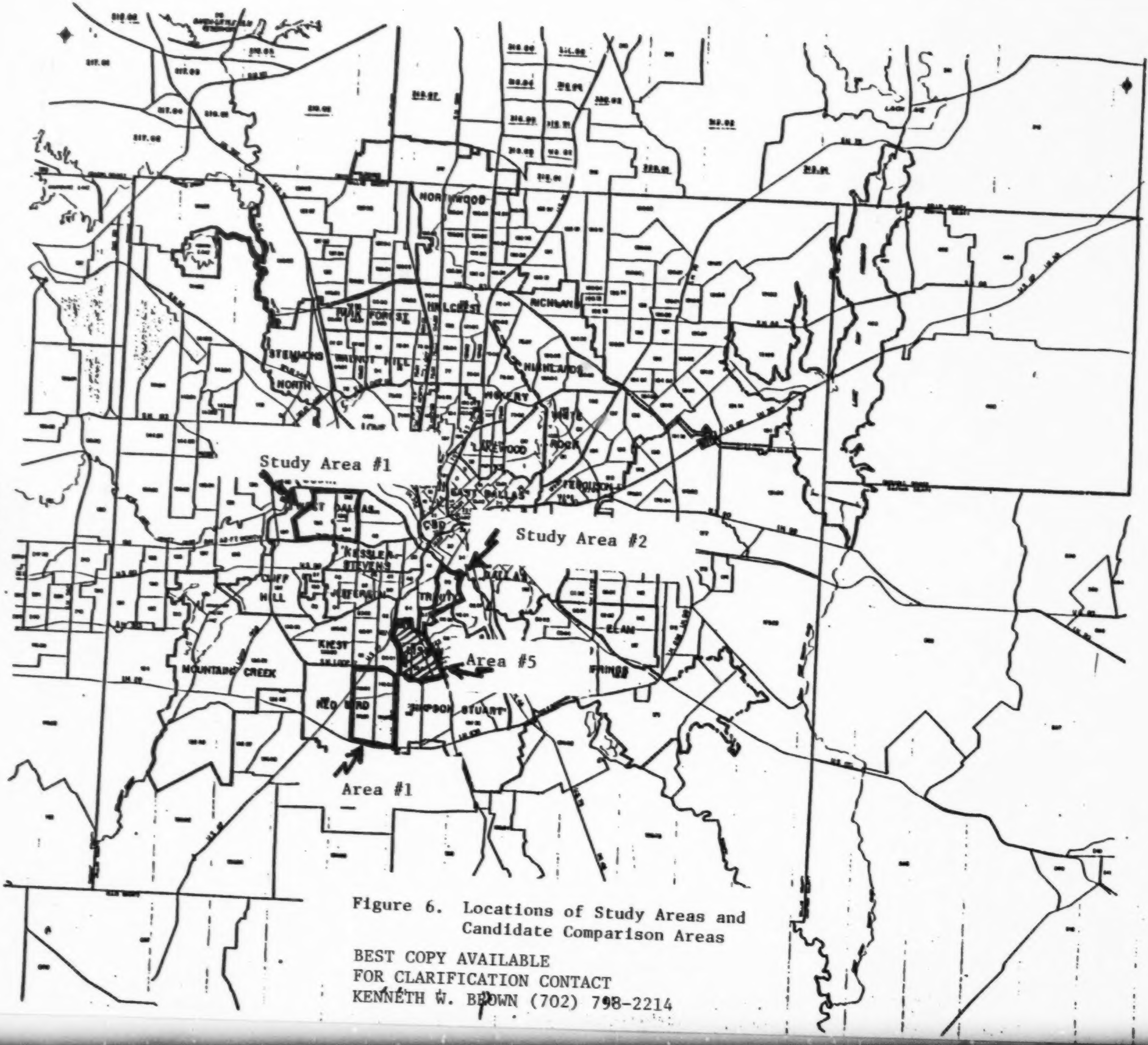
Study Area #1
Study Area #2
Area #5
Area #1
NORTHWOOD
HILLCREST
RICHLAND
PARK FOREST
STEMMONS
WALNUT HILL
NORTH
HIGHLANDS
WHITE ROCK
LAKEWOOD
EAST DALLAS
CBD
WEST DALLAS
KESSLER-STEVENS
CLIFF HILL
JEFFERSON
TRINITY
KIEST
MOUNTAIN CREEK
RED BIRD
SIMPSON STUART
ELAM

Figure 6. Locations of Study Areas and Candidate Comparison Areas

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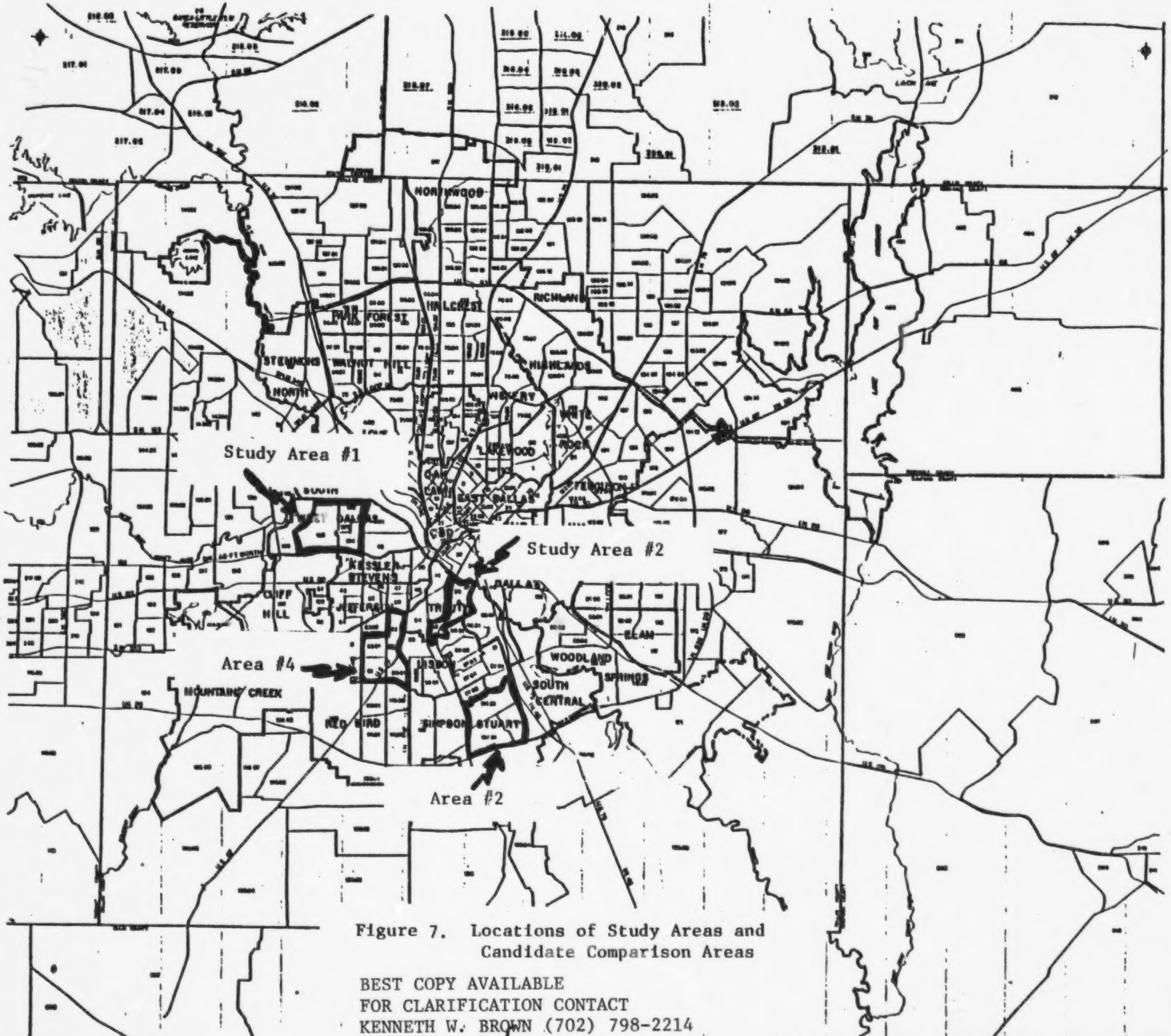
NORTHWOOD
RICHLAND
PARK FOREST
HILLCREST
STEMMONS
WALNUT HILL
NORTH
HIGHLANDS
WHITE ROCK
LAKEWOOD
Study Area #1
EAST DALLAS
FERGUSON
WEST DALLAS
CBD
Study Area #2
KESSLER
STEVENS
CLIFF HILL
JEFFERSON
TRINITY
ELAM
Area #4
LISBON
WOODLAND
SPRINGS
MOUNTAIN CREEK
SOUTH
CENTRAL
RED BIRD
SIMPSON STUART
Area #2

Figure 7. Locations of Study Areas and Candidate Comparison Areas

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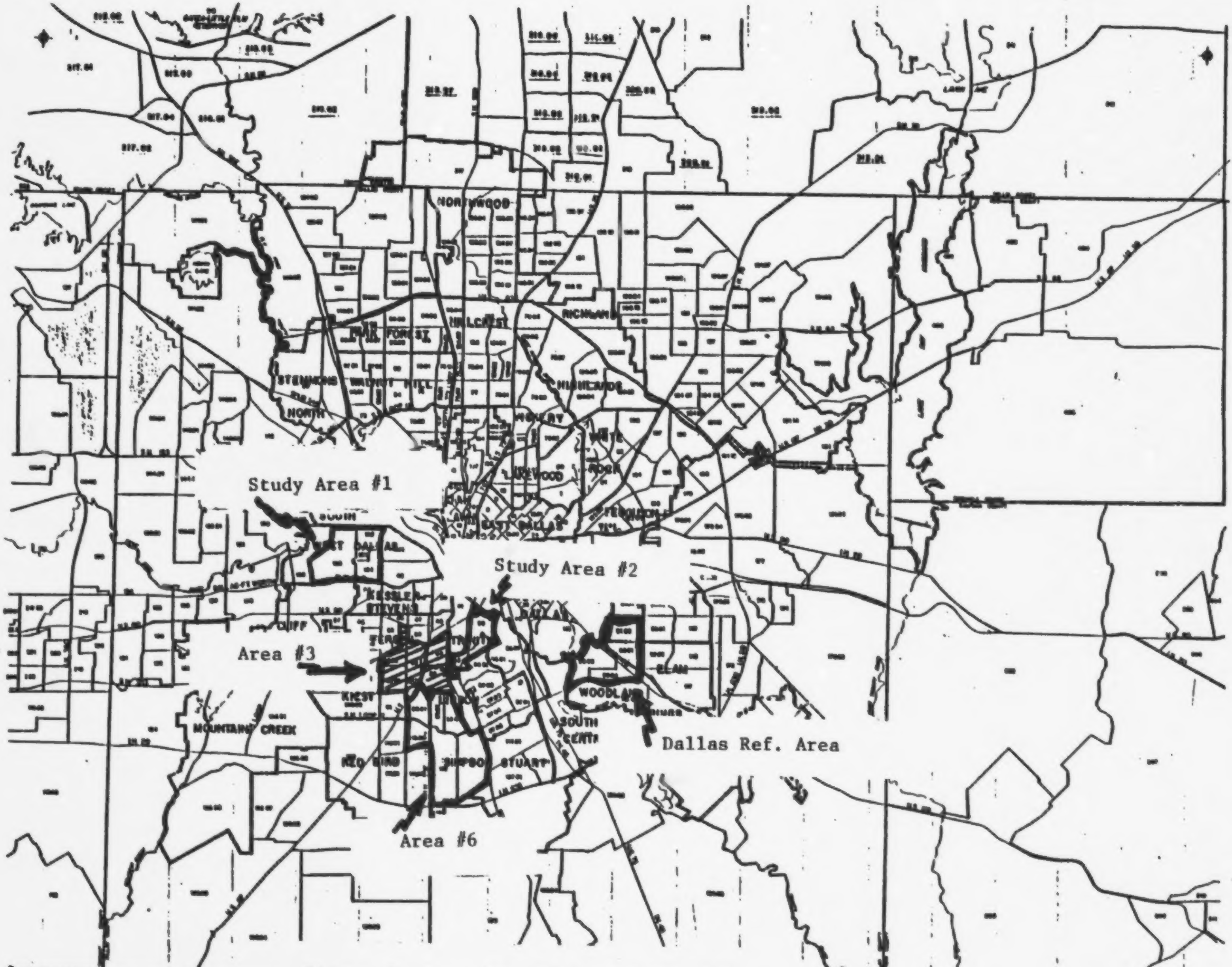
Study Area #1
Study Area #2
Area #3
Area #6
Dallas Ref. Area
NORTHWOOD
HILLCREST
RICHLAND
PARK FOREST
STEMMONS
WALNUT HILL
NORTH
HIGHLANDS
LAKEWOOD
WHITE ROCK
FERGUSON
EAST DALLAS
WEST DALLAS
KESSLER
STEVENS
CLIFF
KIEST
MOUNTAIN CREEK
RED BIRD
STUART
WOODLAND
SOUTH CENTRAL

Figure 8. Locations of Study Areas and Candidate Comparison Areas

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promising of these comparison areas using block level data will be done when Census Maps are provided by the City of Dallas. Because the detailed investigation is time consuming it was felt to be premature at this time.

The statistics on population and housing are based on the 1980 Census of Population and Housing and were compiled from a tape provided by the city of Dallas. The formal Bureau of Census definition of a housing unit is contained as an appendix. The traffic volume was computed as an average of the traffic volume data indicated for sections of roads within the one mile circle. The traffic volume data were for the years 1973 to 1980. These average traffic volumes are only approximate measures because few values existed for any roadway for the same year or for the same section of roadway.

D. Questionnaire Review and Field Training Manual

A minimal instrument consisting of a household roster, demographics, and possibly a few screening and occupation questions will be prepared by CDC. RTI will review the questionnaire primarily for format to assure compatibility with computer entry of the data. Consideration will also be given to both the content and wording of questions.

An instructional field manual will be developed covering household selection and questionnaire administration techniques. The manual will be used at the training session and will be carried into the field by the interviewers for use as a reference. This manual will contain sample list unit sketches, list unit sheets, sample questionnaires, interviewing rules and tips.

E. Field Data Collection Training Session

A training session will be held to instruct the field interviewers in the procedures for identifying selected households and administering questionnaires. Interviewers will be instructed in the proper techniques for administering a questionnaire and will administer several practice questionnaires, receiving feedback from the training instructor.

F. Data Processing

All questionnaires and chemical analysis reports will be logged in upon receipt and manually edited and coded. Questionnaires will be keyed on video display terminals with programmed automatic range and data type edits. Questionnaires will receive a 10% double rekey quality control check. Chemical analysis reports will receive 100% key verification. A limited machine edit of the resulting file will also be conducted.

IV. STATISTICAL ANALYSIS

A. Calculation of Sampling Weights

To properly statistically analyze the data collected in the survey, sampling weights must be calculated based on the individuals probability of selection. The probability of selection ($\pi(i,j,k)$) is given by

$$\pi(i,j,k) = \pi(i,j)\ \pi(k|i,j) \tag{1}$$

where $\pi(i,j)$ is the probability that block j is selected in stratum i and $\pi(k|i,j)$ is the probability of selecting residence k given block j and stratum i. This probability of selection is used for every person residing at the sample residences. The sample weight is the inverse of the selection probability and is given by

$$w(i,j,k) = 1 / \pi(i,j,k) \ . \tag{2}$$

B. Estimation and Testing

The weighted sample mean of stratum i, is calculated by

$$\bar{x}(i) = \frac{\sum_{j=1}^{n(i)} \sum_{k=1}^{m(i,j)} w(i,j,k)\ x(i,j,k)}{\sum_{j=1}^{n(i)} \sum_{k=1}^{m(i,j)} w(i,j,k)} \ . \tag{3}$$

where n(i) is the number of PSUs in stratum i and m(i,j) is the number of preschool children selected in block j of stratum i. It can be shown that $\bar{x}(i)$ is an unbiased estimate of the population mean $\mu(i)$.

To illustrate how one can test the effects of the traffic emissions and smelter emissions we assume the domain means are of the form,

$$\mu(1) = \mu + T + S, \tag{4}$$

$$\mu(2) = \mu + S, \tag{5}$$

$$\mu(3) = \mu + T, \tag{6}$$

and

$$\mu(4) = \mu \tag{7}$$

where T is the contribution to mean lead levels due to traffic, S is the contribution to mean lead levels due to the smelter, and μ is the contribution to mean lead levels due to other sources. Domain one is formed by collapsing strata classified as high for both smelter and traffic emissions. Domain two is formed by collapsing strata classified as high smelter and low traffic. Domain three is formed by collapsing strata classified as high traffic and low smelter. Domain four is formed by collapsing strata classified as low smelter and low traffic. The domain mean estimates, denoted by $\hat{\mu}(i)$, $i = 1, 2, 3, 4$, are obtained by weighted average of the appropriate strata means.

The effect of the smelter and traffic can be tested and quantified. To estimate T, use

$$(\hat{\mu}(1) - \hat{\mu}(2))/2 + (\hat{\mu}(3) - \hat{\mu}(4))/2 \ . \tag{9}$$

To estimate S, use

$$(\hat{\mu}(1) - \hat{\mu}(3))/2 + (\hat{\mu}(2) - \hat{\mu}(4))/2. \tag{10}$$

These estimates can be used to estimate the contribution to the mean lead levels from each source, to compare the relative contribution from each source, and to test if each source has a significant effect on the mean lead levels.

From a statistical view point, including all three study areas is preferable for several reasons: (1) permits estimation of the effect of each smelter, (2) it allows comparisons of the magnitude of the two effects and (3) the test should be able to detect smaller differences. The advantage of using only study area one is that the comparison area has less potential for influence from yet other major lead sources and other factors.

The above test of hypothesis and confidence intervals for the estimates can be constructed for each of the population parameters and media mentioned in section I.D individually using traditional univariate techniques. However, multivariate techniques treating all the observations from each resident as a vector allow simultaneous comparison for the different measurements.

Let X(i,j,k) denote the vector of observations from residence k in block j in stratum i. Let $\bar{X}(i)$ denote the vector of weighted means of these observations from stratum i. $\bar{X}(i)$ is calculated using equation (3) with X(i,j,k) replacing x(i,j,k). Equations (4) - (10) directly generalize to the multivariate context with the scalers be replaced by the vector anologs.

The traffic and smelter effects can be tested in the multivariate setting using the F or Hotellings T^2 distribution (Graybill p.353). Confidence intervals for the traffic and smelter effect estimates can be constructed using the multivariate t distribution (Graybill p.203). Confidence intervals for other comparisons of interest can be tested using "Scheffe's method of simultaneous confidence intervals" (Graybill p.198).

V. QUALITY ASSURANCE

A minimum subsample of 50 specimens from each media should be collected in duplicate in the field. These should be analyzed blind in the laboratory along with other field specimens. This will allow estimation of the chemical analysis precision. Control charts should be maintained by the laboratories including spiked specimens to allow one to control and estimate the accuracy of the measurements. Appropriate procedures for storing and shipping specimens should be investigated and followed. The inclusion of field blanks and spikes is a way of assessing the potential magnitude of contamination and loss resulting from field procedures.

VI. REFERENCES

Graybill, FA. 1976. Theory and Applications of the Linear Model. Duxbury Press, North Scitute, Massachusetts.

APPENDIX A

CENSUS DEFINITION OF A HOUSING UNIT

2.FF Housing Unit

A housing unit is a house, an apartment, a group of rooms, or a single room occupied as a separate living quarters, or if vacant, intended for occupancy as a separate living quarters.

1. "Separate living quarters" are those in which:

   a the occupant(s) live and eat separately from other person(s) in the building, and

   b the occupant(s) have direct access from the outside of the building or through a common hall (as in an apartment building, etc.).

2. If vacant, the criteria of separateness and direct access apply to the intended occupants.

3. If occupied, a tent, houseboat, lean-to, etc. may be a housing unit.

4. If a living quarters contains nine or more persons unrelated to the resident who owns or rents the quarters, or ten or more unrelated persons, it is not a housing unit, but a group quarters.

APPENDIX E

QA/QC PROCEDURES FOR THE DALLAS LEAD STUDY

QA/QC PROCEDURES FOR THE DALLAS LEAD STUDY

Introduction

The EMSL-LV was assigned responsibility for collection and analysis of soil samples and for analysis of dust swipe samples. In response, protocols were developed to specify the collection of soil samples, the operations of a sample bank for identifying and transferring soil and dust samples under chain-of-custody procedures, and the analytical methods to be used for soil and for dust samples.

To handle the large number of samples efficiently and economically and yet achieve results of high quality, the protocols included a significant number of QA/QC samples and procedures. These would check the adequacy of the specified analytical procedure as well as the adequacy of decontamination of the sample collection and preparation apparatus. A description of the QA/QC procedures, the data validation method, the results obtained, and the overall uncertainty of the analytical data are explained in the following sections.

Methods

The methods used are set forth in detail in the analytical protocols for soils and for dusts. Criteria for data acceptability were specified in the QA Project Plan. Briefly, each group of samples was analyzed along with the specified QA samples, the results were sent to EMSL-LV for validation, and finally the valid results were sent to RTI for entry into the data base.

Because different sample collection methods were used for soil and dust samples, the QA procedure also differed for the two types of samples. The procedures used are outlined below.

QA/QC PROCEDURES FOR DUST SAMPLES

Procedure	Comment
1. Field Blank	Package of swipes taken by field personnel to the sampling site. Opened and exposed during sampling, but not used to collect samples. Checks for accidental and incidental contamination.
2. Reagent Blank	One for each 20 samples. Five ml of distilled water extracted and analyzed like a dust sample to check for reagent contamination level.
3. Calibration Check Standard	One for each 20 samples. An appropriate dilution of EPA certified trace metal standard analyzed to check instrument calibration.
4. Spiked Extract	One for each 20 samples. A portion of the sample extract spiked with NBS Lead Nitrate standard to detect extract matrix effects on recovery of known added analyte.
5. Split Extract	One for each 20 samples. A separate aliquot of the sample extract is analyzed to check instrument and injection reproducibility.

QA/QC PROCEDURES FOR DUST SAMPLES (cont'd)

Procedure	Comment
6. Laboratory Control Standard	One for each 20 samples. An NBS Urban Particulate sample carried through the analytical procedure to check for overall method bias.
7. Duplicate Sample	One for each 20 samples. A separate sample taken within 50 cm of original sample to determine total random error in collection and analysis.

Two other procedures used were: 1) re-extraction analysis to determine the efficiency of the initial extraction, and 2) analysis of residual sample extracts for 44 samples by a referee laboratory to detect any unrecognized bias.

QA/QC PROCEDURES FOR SOIL SAMPLES

Procedure	Comment
1. Field Blanks	One for each sampling team per day. A sample container filled with distilled, de-ionized water, exposed during sampling then analyzed to detect accidental or incidental contamination.
2. Sample Bank Blanks	The field blank, about 40% of them, passed through the sample preparation apparatus, after cleaning, to check for residual contamination.

QA/QC PRCOEDURES FOR SOIL SAMPLES (cont'd)

Procedure	Comment
3. Reagent Blank	Same as above. One for each 20 samples to check reagent contamination level.
4. Calibration Check Standard	Same as above. One for each 20 samples to check instrument calibration.
5. Spiked Extract	Same as above. One for each 20 samples to check for extract matrix effects on recovery of known added analyte.
6. Spiked Sample	One for each 20 samples. A separate aliquot of the soil sample spiked with NBS Lead Nitrate to check for soil and extract matrix effects on recovery.
7. Total Recoverable	One for each 40 samples, a second aliquot of the sample is digested by a more vigorous method to check the efficacy of the protocol method.
8. Laboratory Control Standard	One for each 20 samples. A sample of NBS River Sediment carried through the analytical procedure to determine overall method bias.
9. Re-extraction	One for each 20 samples. A re-extraction of the residue from the first extraction to determine extraction efficiency.

QA/QC PROCEDURES FOR SOIL SAMPLES (cont'd)

Procedure	Comment
10. Split Extract	Same as above. One for each 20 samples to check injection and instrument reproducibility.
11. Triplicate Sample	One for each 20 samples. The prepared sample is split into three portions to provide blind duplicates for the analytical laboratory and a third replicate for the referee laboratory to determine interlab precision.
12. Duplicate Sample	Same as above. One for each 20 samples to determine total random error.

Data Validation

The analytical laboratories (City of Dallas for the dust swipe samples and GCA Corp. for soil samples) forwarded the analytical results on groups of 20 samples and accompanying QC results to EMSL-LV using the appropriate forms specified in the analytical protocol.

Validating each set of 20 samples of either soil or dust consisted of three checks based on the accompanying QC results. These checks were:

1. Reagent Blanks - Either <0.25 μg/ml for soil or <0.1 μg/ml for dust, based on use of a different absorbance wavelength for each type of sample;

2. Calibration Check Standards - Recovery must be between 95% and 105% of the known value for either the first analysis or the first re-check analysis;

3. Laboratory Control Standard - Recovery must be between 90% and 110% of the known value for either the first analysis or the first re-check analysis.

All data sets received from the analytical laboratories met the above criteria and were accepted as valid data sets. The results were assembled in numerical order according to categories of dust samples, grid soil samples, or house soil samples, and were then forwarded to RTI for entry into the data base.

Results

The results produced by any sampling and analyzing system are affected by two types of error; random and systematic. The accuracy of any one result, then, is a function of the bias (due to systematic error) and precision (due to random error) of the collection and analysis methodology. Bias has at least two components, associated with extraction and instrument efficiency, and is assessed by the mean recovery of Calibration Check Standards and Laboratory Control Standards (LCS). The LCS checks overall bias for the system; the Calibration Check Standard determines the instrumental bias.

Total random error can be assessed by analyzing duplicate samples, but it includes errors due to sample collection, sample homogeneity, sample extraction, sample composition (matrix effects) and instrumental reproducibility. These errors can be evaluated by the use of the other QC procedures stated above and are assessed by calculating the standard deviations of the various analyses.

The accuracy of analysis, i.e., bias and precision, are evaluated separately below for the two types of samples, using the following equations:

$$\text{Recovery} = \text{Amount Found/Known Amount} \qquad (1)$$

$$\text{Bias (B)} = \text{Recovery} - 1 \qquad (2)$$

Difference (D) = $|x_1 - x_2|$ where x_1 and x_2 are the analytical results of paired analyses and the average difference is:

$$\bar{D} = \frac{\sum_n |x_1 - x_2|_i}{n} \qquad (3)$$

and the precision is:

$$S\bar{x} = \text{Precision} = 0.8862\ \bar{D} \qquad (4)$$

where 0.8662 converts the range of two results to the standard deviation.

If component errors are used to assess total random error, then

$$\bar{D} = (\bar{D}_1 + \bar{D}_2 + \ldots,)/n \text{ and}$$

$$S\bar{x} = \text{Precision} = 0.8862\ (\bar{D}_1^2 + \bar{D}_2^2 + \ldots)^{1/2} \qquad (5)$$

Equation (3) is suitable for use on results where the concentration varies over a very narrow range. If the concentrations found vary by an order of magnitude or more, then the difference should be normalized by dividing by the average of the two values. Then, the precision is expressed as the coefficient of variation (CV) which is $S/\bar{x}$.

$$\bar{D}_n = (\sum_n |x_1 - x_2|_i) \div (\frac{x_1 + x_2}{2})$$

$$= \sum_n 2|x_1 - x_2|_i/(x_1 + x_2) \qquad (6)$$

$$CV = 0.8862\ \bar{D}_n \qquad (7)$$

Dust Samples

The results for the dust swipe samples analyzed by the City of Dallas Laboratory indicated that more than 95% of the samples contained lead in concentrations near the limit of detection for the AAS method used. The results of the QC samples were as follows:

QC Sample	No.	Mean	S	Range
Calibration Check Standard	45	100.7%	2.4%	96.0 - 105%
Laboratory Control Standard	46	97.0%	2.7%	93.1 - 102.3%
Field Blank ($\mu g/cm^2$)	53	0.036	0.041	0 - 0.21 net
Reagent Blank ($\mu g/cm^2$)	35	0.063	0.021	0.03 - 0.13
Split Extract ($\mu g/cm^2$)	45	0.082	0.073*	0 - 0.22
Spiked Extract	57	104.1%	6.2%	92 - 120%
Duplicate Sample ($\mu g/cm^2$)	48	0.042	0.037*	0 - 0.27

* Calculated as 0.886 x mean difference.

Bias--The results of the Calibration Check Standards indicate a stable analytical instrument with no bias. The LCS results indicate a small negative bias in the method due mostly to the extraction of the analyte from the sample. Using equation (2), the bias is:

$$B = 0.97 - 1 = -0.03$$

Precision--Since all results covered a range of less than a factor of 10, equation (4) can be used to estimate precision. Thus, from duplicate sample analyses:

$$Sx = 0.8862\ \overline{D} = 0.8862\ (0.042) = 0.037\ \mu g/cm^2$$

The random errors that contribute to this overall error can be estimated by the use of equation (5) as follows:

$$\begin{aligned} Sx &= (0.041^2 + 0.021^2 + 0.073^2 + 0.062^2)^{\frac{1}{2}} \\ &= 0.107 \end{aligned}$$

This result suggests that extract matrix effects and instrumental reproducibility errors, not sampling error, control the precision of analysis. This is probably to be expected given the contaminant levels found in the medium (dust swipes) examined and, perhaps, a fairly even distribution of airborne lead. From the above calculations, the precision is estimated as:

$$\text{Precision} = 0.107 \quad g/cm^2$$

The random error due to the field blanks is substantial, 0.041 $\mu g/cm^2$, but this is apparently due to the collection medium as the standard deviation of the analysis of six unopened cotton swabs was 0.07 (range of net results, 0 - 0.19).

A check on possible analytical bias was performed by submitting 44 dust swipe extracts to the referee laboratory for analysis. The referee laboratory results were much higher than the Dallas Laboratory results for all samples except the few having the highest sample concentration. Since contamination of the samples was suspected, and since this would affect low-level samples more than those with high levels, 13 of the residual extracts were returned to Dallas for re-analysis. The standard deviation of the paired analyses on these 13 extracts was 0.059 μg/ml, indicating negligible bias between the laboratories.

These results suggest that contamination of the extracts occurred after the first analysis and that extreme precautions must be taken with extracts containing lead concentrations near the limit of detection to prevent accidental contamination during transfer or storage.

Soil Samples

The results for the soil samples analyzed by the GCA Corp. ranged from near the limit of detection, 5 μg/g, to about 10,000 μg/g. This limit of detection, approximately 0.25 μg/ml, was tested on about 10 blank analyses using the more sensitive absorbance wavelength for lead on the AAS. The result was less than 0.1 μg/ml, or 2 μg/g, for sample analysis. This suggests that most of the blank analyses were less than 2 μg/g, but this cannot be stated with any confidence. The results of the QC analyses were as follows:

QC Sample	No.	Mean	S	Range
Calibration Check Standard	150	101.5%	2.6%	93.6 - 110%
Laboratory Control Standard	147	101.2%	4.1%	90.3 - 109.9%
Field Blank	76	<0.25		
Sample Bank Blank (μg/ml)	77	<0.25		
Reagent Blank (g/ml)	148	<0.25		<0.1 - <0.25
Re-extraction Analysis	17	1.7%	1.4%	0.2 - 3.8%
Total Recoverable	144	99.8%	8.0%	78.2 - 122.1%
Split Extract (CV)	147	0.0089	0.0079	0 - 0.081
Spiked Extract	147	99.4%	5.0%	87 - 111%
Spiked Sample	147	100.4%	5.1%	87.3 - 110.5%
Duplicate Aliquot (CV)	134	0.053	0.047	0 - 0.269
Duplicate Sample (CV)	129	0.189	0.168	0.002- 0.931
Triplicate Analyses (CV)	220	0.144	0.128	0 - 0.826

Bias--The percent recoveries indicated above for the Calibration Check Standards and LCS's suggest a positive bias for the protocol method of soil analysis, due principally to instrument reproducibility. The result, using equation (2), is:

$$\text{Bias} = \text{Recovery} - 1 = 1.012 - 1 = 0.012$$

Precision--The recovery of analyte by the protocol method compared to a "total" recoverable method was essentially equal and re-extraction of th[illegible]sidue left from the initial extraction indicated an additional 1.7 ± 1.4% recovery, also essentially equivalent. Furthermore, the results of the three types of blank analyses indicate no measurable contamination from reagents, sample collection, or sample preparation. The remaining random errors are evaluated below. Because of the wide range of concentration of lead in the samples, the coefficient of variation is used, equation (7).

Precision (total random error) from Duplicate Sample analysis:

CV = 0.168 or 16.8% of sample concentration

The component random errors, summed as per equation (5), are:

$$Sx = (0.0079^2 + 0.05^2 + 0.051^2 + 0.047^2)^{\frac{1}{2}} = 0.085$$

These random errors suggest that reproducibility errors (0.0079) are small and that extract matrix, sample matrix, and sample homogeneity errors are equivalent. The sum of these errors is about half the total random error so the sampling error is essentially equal to all other errors combined.

Interlaboratory precision as calculated from the results of triplicate analyses, using equation (7), is:

Precision = CV = 0.128 or 12.8% of sample concentration

Uncertainty

The data for bias and precision can be combined to yield the uncertainty for any reported concentration by use of the following equation:

$$U = (1 + B \pm 2C) \tag{8}$$

where B is the bias, C is the standard deviation or coefficient of variation as appropriate, and 2 converts these to the 95% confidence limits.

For dust swipe samples, using equation (8) and the bias (-0.03) and precision (0.107) derived above, the 95% confidence bounds on a reported value, X, are:

Dust result will lie between 0.896X and 1.044X $\mu g/cm^2$.

For soil analyses, using equation (8) and the bias (0.012) and CV (0.168) derived above, the 95% confidence bounds on a reported value, X, are:

Soil result will lie between 0.676X and 1.348X $\mu g/g$.

APPENDIX F

STATISTICAL ANALYSIS OF QUALITY CONTROL DATA

STATISTICAL ANALYSIS OF QUALITY CONTROL DATA

The 50 percent level of quality control implemented during the Dallas Lead Study provided a substantial data base for statistical analysis. Each set of 20 field sample analyses was monitored by the analysis of 12 quality control samples. The resultant data set of each program quality control category was individually subjected to statistical analysis. Table 5-1 summarizes the quality control analyses performed, their means and standard deviations. Where applicable, means are expressed as a percentage of the expected value to afford continuity and facility of comparison.

Initially the individual frequency of each quality control category was 1 in 20, with the exception of the "total recoverable" analysis which was 1 in 10. The reextraction and "total recoverable" analyses were utilized to monitor the extraction efficiency of the protocol procedure. After consistent evidence of 100 percent extraction of the lead by the protocol procedure, as supported by 16 of 17 analyses performed, the reextraction analysis was terminated. The frequency of the "total recoverable" analysis was reduced to 1 in 40 after the analysis of 94 samples indicated no significant bias towards either methodology.

To eliminate unintentional biasing of data, the quality control samples were identified and analyzed according to the sequence described by the provided EPA protocol, Appendix A. The initial sample for each sequence was chosen by a random generator.

The results of the statistical examination of each quality control sample category are summarized below:

1. Method (Reagent) Blank--The reagent blank was prepared by carrying reagents alone through the digestion procedure. Figure 5-1 represents the frequency distribution of the resultant program reagent blanks. The calculated mean (0.007 μg/ml) is well below the analytical detection limit of 0.25 μg/ml. It should be noted that the 3s value is at the limit of detection. It may be concluded that the mean is not significantly different from zero and that no appreciable contamination was realized during the digestion of the program samples.

TABLE 5-1. SUMMARY OF STATISTICAL ANALYSIS OF QUALITY CONTROL DATA

Quality control parameter	n	-3	$\sqrt{B_1}$	$\bar{x}$	1s	2s	3s	$s_{\bar{x}}/\bar{x}$ (%)
Reagent Blank	148	0.0003	0.801	0.007	0.08	0.16	0.24	—
Field and Sample Bank Blank	150	0.0003	0.630	0.01	0.09	0.18	0.27	--
Split Extract (as % original extract)	148	2.24	0.549	100.1	2.0	3.9	5.9	2.0
Total Recoverable (as % protocol extract)	147	367	0.806	100.4	8.3	16.5	24.8	8.2
Reextraction	17	—	—	99.8 (% recovery in original)	0.7	1.4	2.8	0.7
Spiked Sample (As % recovery of spike)	148	16.2	0.340	100.3	5.2	10.4	15.6	5.2
Spiked Extract	131	0.001	0.141	7.95 (99.3% recovery of 8.00 μg Pb/ml spike)	0.37	0.75	1.1	4.7
Calibration Check Standard	450	0.001	0.481	9.96 (99.6% recovery of true value)	0.18	0.36	0.54	1.8
Calibration Control (WP 475 Conc 3):								
2.50 μg Pb/ml	74	0.0001	0.608	2.54 (101.6% recovery of true value)	0.07	0.14	0.21	2.9
5.00 μg Pb/ml	57	0.00002	0.144	5.09 (101.8% recovery of true value)	0.10	0.20	0.30	2.1
Blind Split Samples	135	111	0.466	-0.32%	8.0	16	24	—
NBS SRM 1645	148	-9846	0.654	726 (101.6% recovery of true value)	28	56	84	3.9

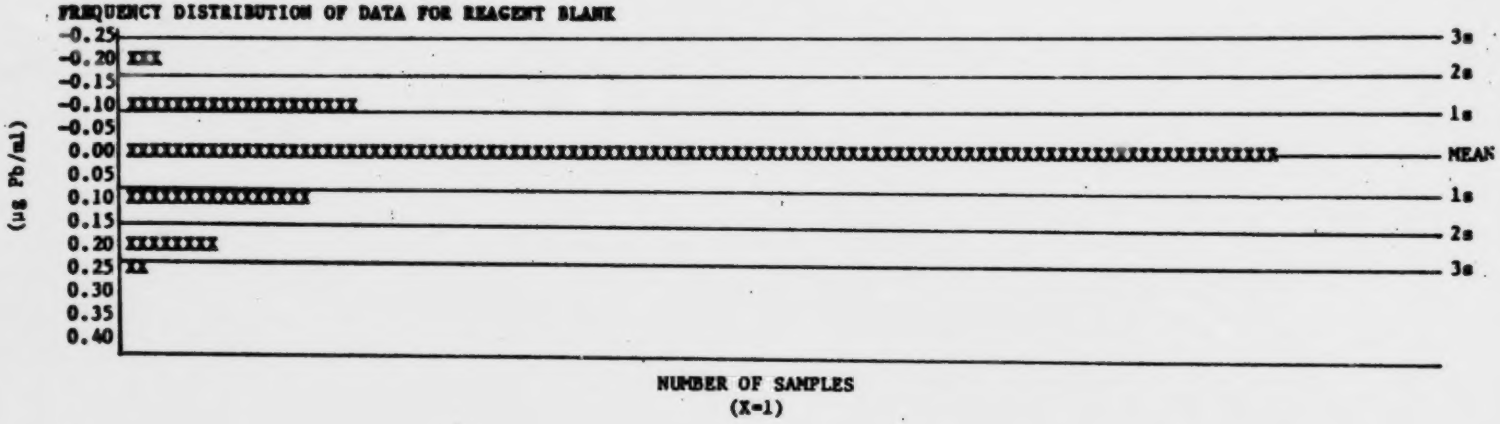

Figure 5-1. Frequency distribution of data for reagent blanks.

2. Field Blank and Sample Bank Blank--In order to monitor possible contamination of samples during sampling efforts, preliminary sample preparation, and shipping, field blanks and sample bank blanks were analyzed. Field blanks, deionized water carried into the field, and sample bank blanks were carried through grinding, sieving, and transportation procedures identical to the soils. The calculated mean (0.014 μg/ml) was again well below the analytical detection limit. Again the 3s value was at the analytical limit of detection. The frequency distribution of this data is presented in Figure 5-2.

3. Split Extract--The split extract constituted the tenth analysis subsequent to that of the parent extract in the analytical sequence. Split extract analysis was performed to estimate intralaboratory precision. The frequency distribution for the split extract data set, expressed as percent recovery of the original extract concentration, is presented in Figure 5-3. The mean recovery was calculated to be 101.6 percent with a standard deviation of 2.0. 96 percent of the data were within the ±3s window.

4. Spiked Extract--The spiked extract quality control sample was utilized to identify matrix interferences. The average recovery of a 200 μg total (8.00 μg/ml additional to extract concentration) lead spike was 99.3 percent with a standard deviation of 0.37 μg Pb/ml. This would indicate that no significant matrix interference was present for soils of this location carried through the protocol digestion. The frequency distribution of the data expressed as μg/ml Pb is presented in Figure 5-4. The index of skewness indicates a confidence level of better than 95 percent.

5. "Total Recoverable"--As indicated the "total recoverable" methodology involved the more rigorous HNO_3/hydrogen peroxide digestion. This quality control analysis was utilized to monitor the efficiency of the protocol procedure. The "total recoverable" (TR) data were compared to the data resultant from the protocol technique for the identical sample. The percent recovery lead was calculated by:

   % recovery = (concentration TR/concentration original) * 100

   The average recovery was determined to be 100.4 with a standard deviation of 8.3. No bias was evident towards either procedure. The frequency distribution of this data set is illustrated in Figure 5-5. 94 percent of the data set were within 2s.

6. Reextraction--17 samples were carried through the reextraction procedure. The mean percent recovery of lead in the original extract was 99.8. The standard deviation was calculated to be 0.7 percent.

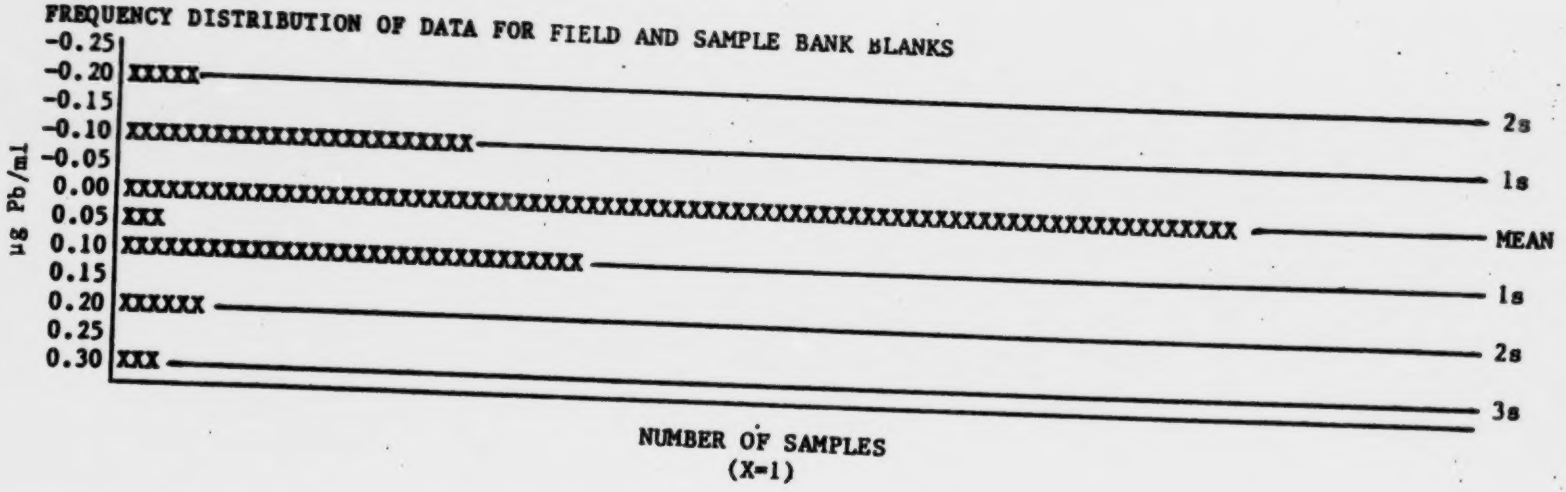

Figure 5-2. Frequency distribution of data for field and sample bank blanks.

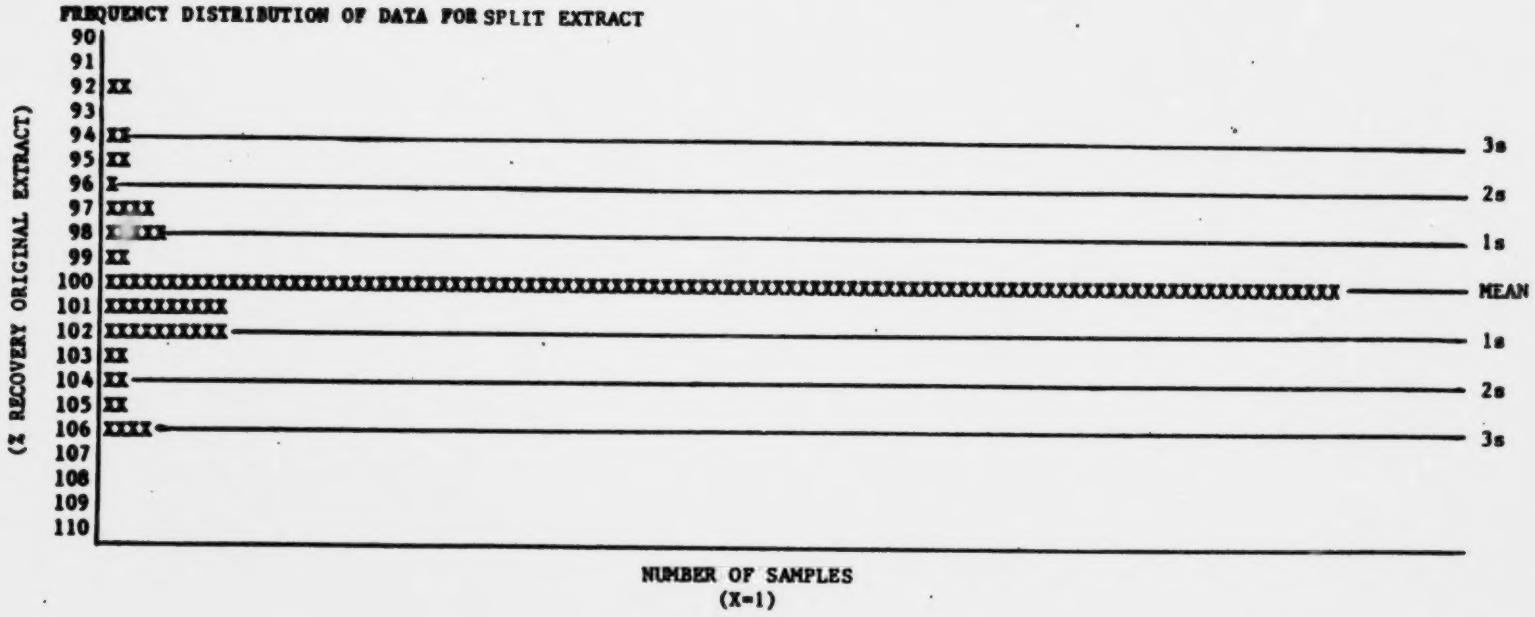

Figure 5-3. Frequency distribution of data for split extract.

```
FREQUENCY DISTRIBUTION OF DATA FOR  SPIKED EXTRACT AT 8.00 µg Pb/ml SPIKE LEVEL
7.05|
7.10|
7.15| X
7.20|--------------------------------- 2s
7.25| XX
7.30| XXX
7.35| XXXX
7.40| XXXXX
7.45| XXX
7.50| XXXXX
7.55|
7.60| XXXXX --------------------------- 1s
7.65| XXX
7.70| XXXXXXXXX
7.75| XXXXXXXX
7.80| XXXXXXX
7.85| XXXX
7.90| XXXXXX
7.95| XXXXX---------------------------- MEAN
8.00| XXXXXXXXXXX
8.05| XXXXX
8.10| XXXX
8.15| XXX
8.20| XXXXXXX
8.25| XXX
8.30| XXXX----------------------------- 1s
8.35| XXXX
8.40| XXXXXX
8.45| XXX
8.50| XXX
8.55| XXX
8.60| XXX
8.65| X
8.70| X -------------------------------- 2s
8.75|
8.80|
8.85|
8.90|
8.95|__________________________________________________

                   NUMBER OF SAMPLES
                         (X=1)
```

SPIKED EXTRACT ÷ :GINAL EXTRACT (µg Pb/ml)

Figure 5-4. Frequency distribution of data for spiked extract at 8.00 µg Pb/ml spike level.

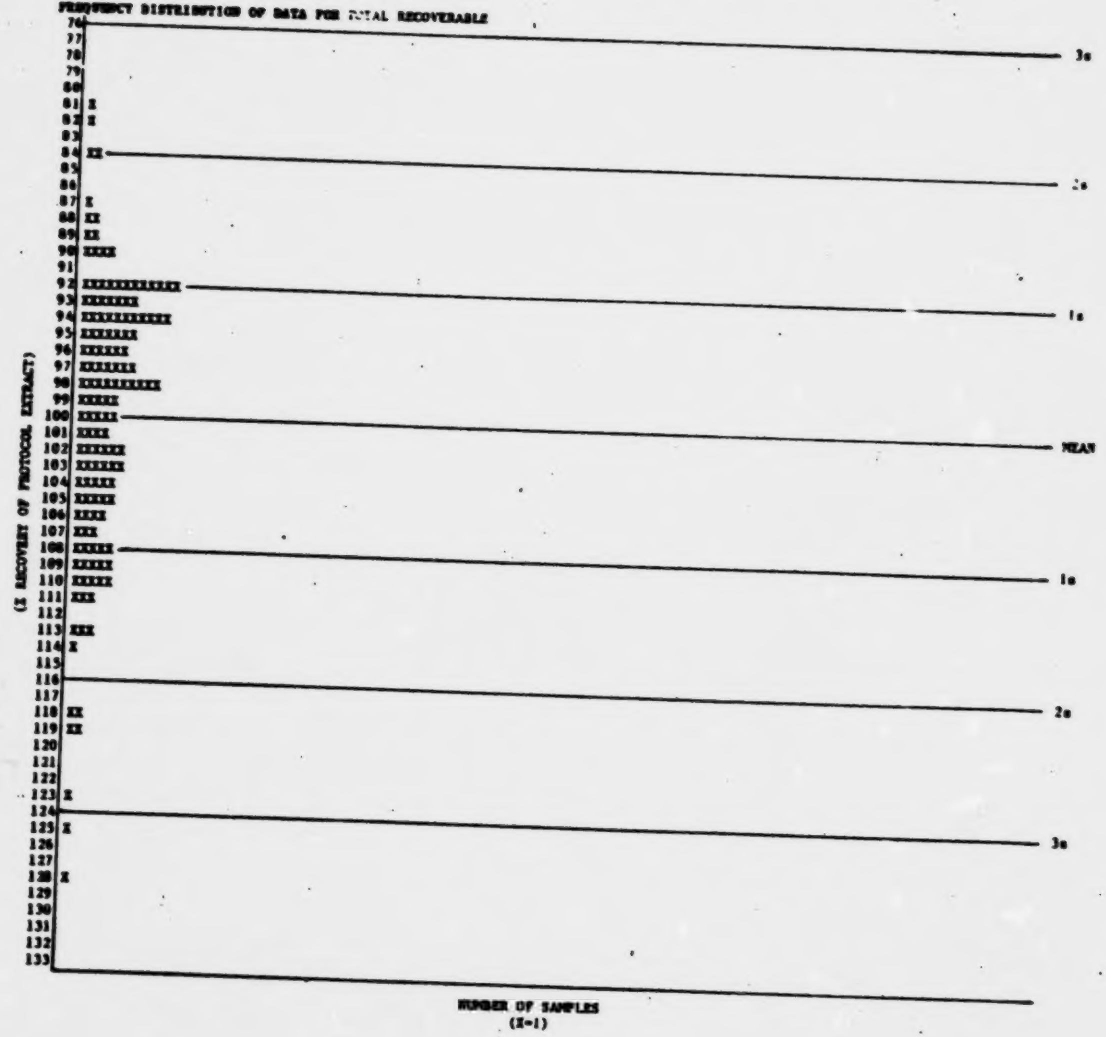

Figure 5-5. Frequency distribution of data for total recoverable.

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7. Spiked Sample--Spiked sample analysis was utilized to indicate lead loss during the digestion procedure. The quality control samples were spiked with 3500 μg total Pb prior to any additon of reagents. The average recovery of the spike was determined to be 100.3 percent with a standard deviation of 5.2 percent. This would indicate no lead loss during digestion either to volatilization or precipitation. The index of skewness places the confidence level at between 95 and 99 percent. The frequency distribution of spiked sample data is illustrated in Figure 5-6.

8. Calibration Check Sample--The atomic absorption spectrophotometers were calibrated at a minimum of once per day. The absorbances of calibration standards at the following levels were utilized for determination of sample concentration; 0, 0.5, 1, 2, 5, 10, 15, 20, and 25 μg Pb/ml. After each tenth analysis in the sequence a calibration control sample, 10 μg Pb/ml, was analyzed to verify calibration. The overall mean was 9.96 μg/ml with a standard deviation of 0.18. Figure 5-7 demonstrates the frequency distribution of the data. 74, 94, and 100 percent of the data fell within 1s, 2s, and 3s limits, respectively.

9. Calibration Control Sample--The calibration control sample was intended as an independent source for the validation of the concentration of the calibration standards and therefore the soil samples themselves. Dilutions of WP475 concentrate No. 3 (prepared by EMSL-CINN) were made to provide 2.50 and 5.00 μg Pb/ml control samples. At least one of these solutions was analyzed at the end of calibration and each analytical sequence. The average percent recoveries of the expected 2.50 μg/ml and 5.00 μg/ml were 101.6 and 101.8 percent, respectively. The data frequency distributions are presented in Figures 5-8 and 5-9. The index of skewness indicates a better than 95 percent confidence at the 5.00 μg Pb/ml level and between 95 and 99 percent confidence at the 2.50 μg Pb/ml level.

10. Blind Split Sample--One in 20 samples were split by the Dallas Sample Bank prior to submittal to the laboratory as two distinct samples. The identity of these samples was not available to laboratory personnel until all final data had been transmitted to EMSL Las Vegas. These data pairs provide a rugged test of the precision of the overall preparative and analytical protocol.

    The percent difference of the data pair was calculated as;

$$[2(X_1 - X_2)/(X_1 + X_2)] * 100$$

    The mean distribution is -0.32 percent, where the sign is determined by the arbitrary assignment of X_1 and X_2. The frequency distribution of the data is illustrated in Figure 5-10. The standard deviation is 8.0 percent. These results demonstrate a remarkable precision in both sample homogenization and analysis.

FREQUENCY DISTRIBUTION OF DATA FOR SPIKED SAMPLE

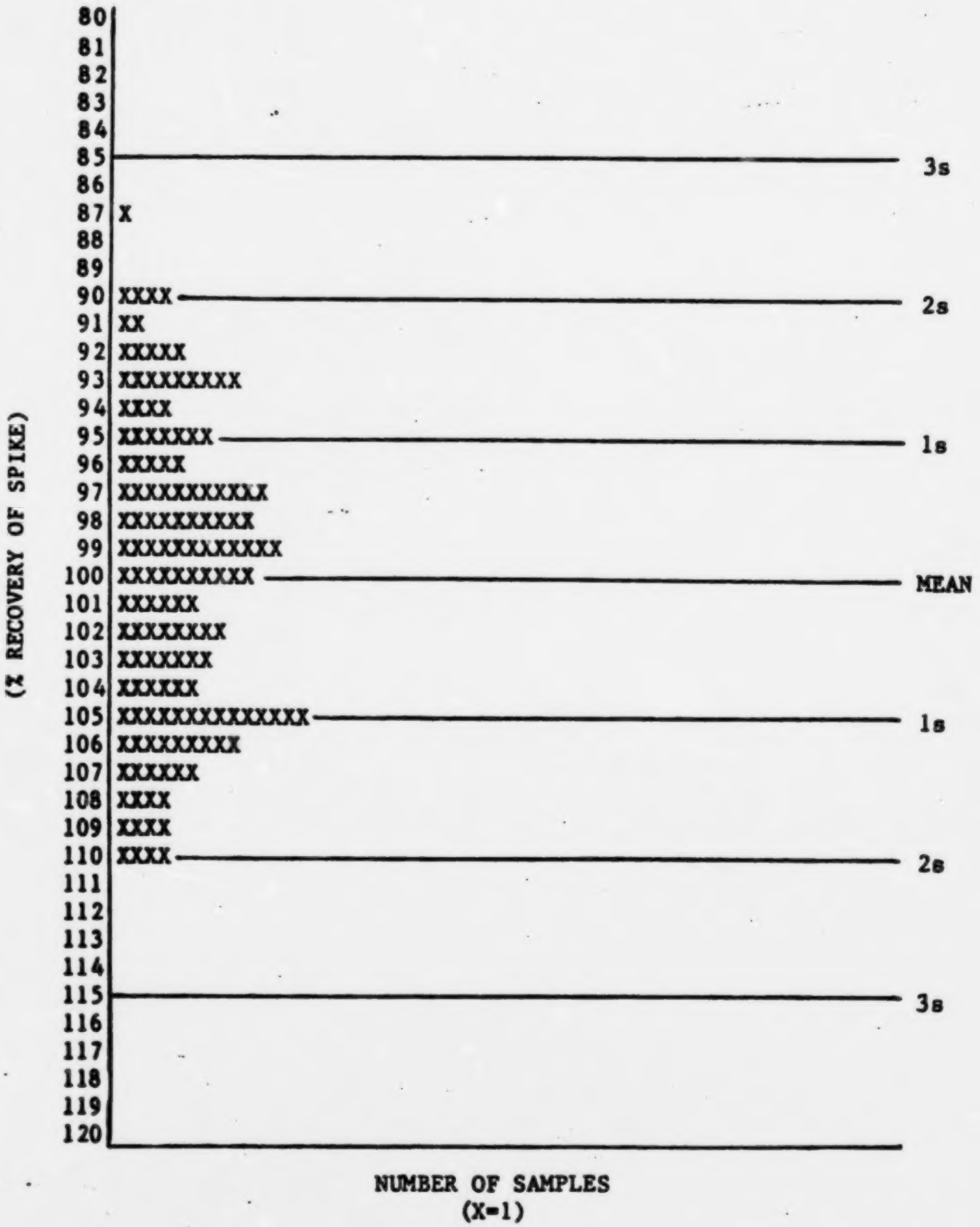

Figure 5-6. Frequency distribution of data for spiked sample.

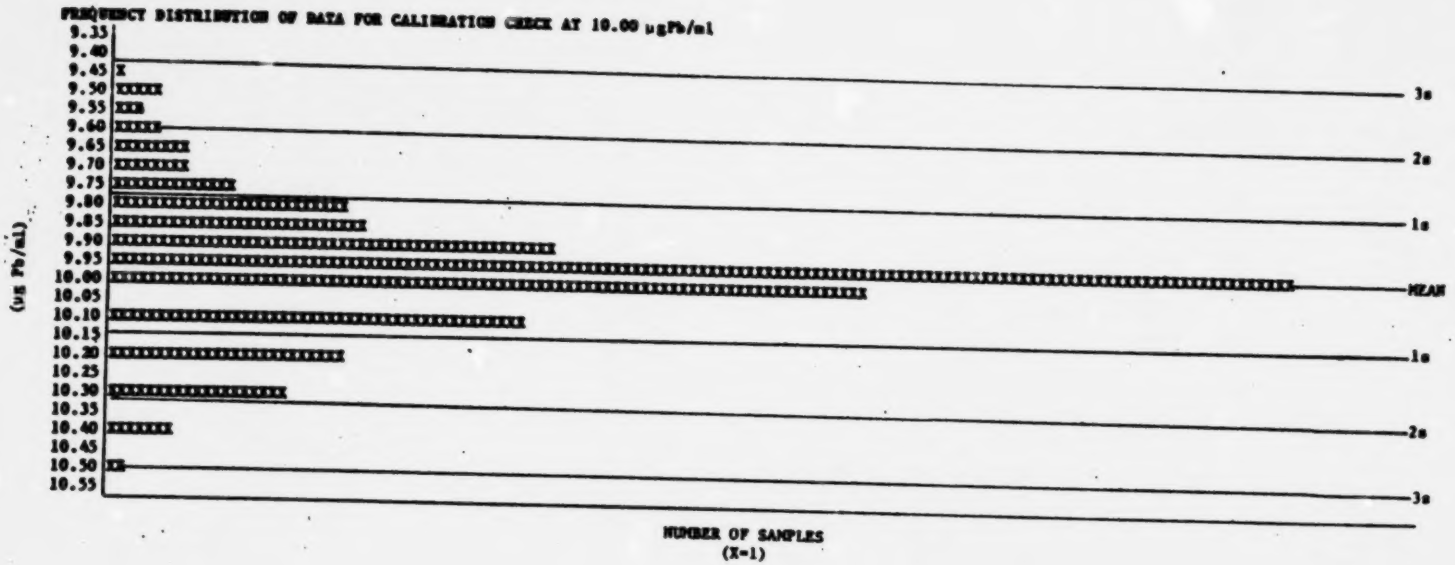

Figure 5-7. Frequency distribution of data for calibration check at 10.00 µg Pb/ml.

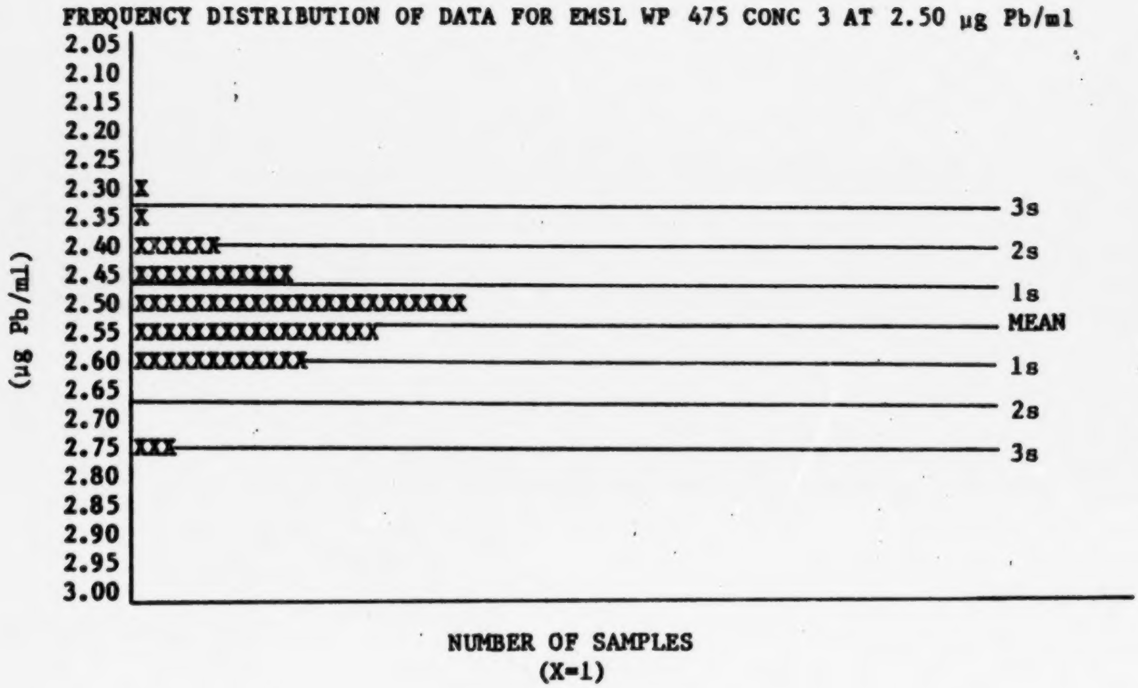

Figure 5-8. Frequency distribution of data for EMSL WP 475 conc 3 at 2.50 μg Pb/ml.

FREQUENCY DISTRIBUTION OF DATA FOR EMSL WP 475 CONC 3 AT 5.00 μg Pb/ml

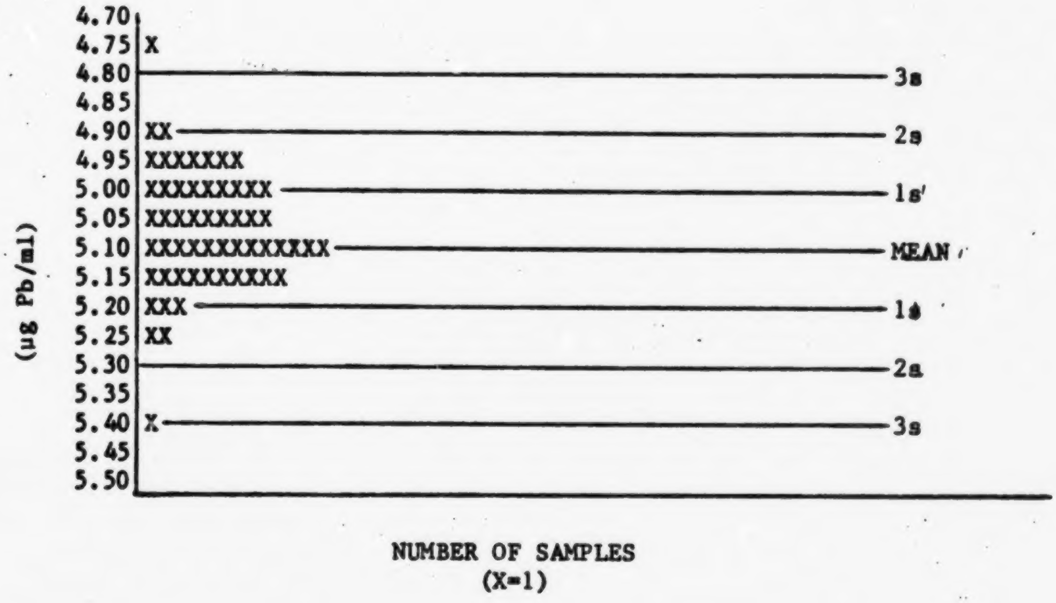

Figure 5-9. Frequency distribution of data for EMSL WP 475 conc 3 at 5.00 μg Pb/ml.

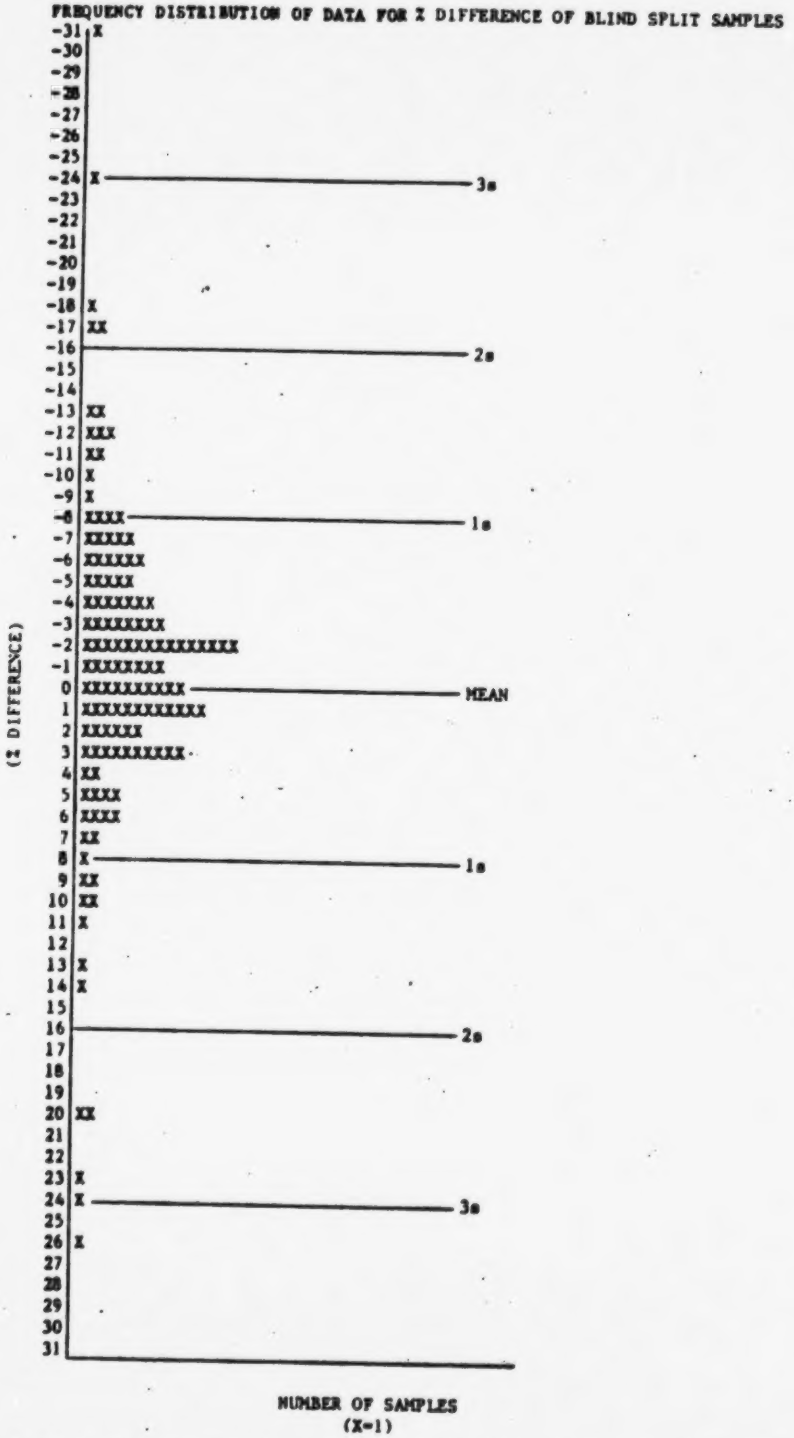

Figure 5-10. Frequency distribution of data for % difference of blind split samples.

10. Laboratory Control Sample Analysis—The analysis of NBS SRM 1645, river sediment, was conducted to monitor the extraction of a known quantity of lead from a matrix similar to the program samples. 5-g aliquots were extracted to mimic sample preparation and analysis. Approximately halfway through the program it became necessary to reduce the aliquot to 1 g to assure sufficient reserve to complete the program.

Statistical examination of the entire data set exhibited a bimodal distribution, Figure 5-11. The calculated mean was 726 μg Pb/g with a standard deviation of 28 μg/g. The analytical mean determined by the protocol procedure was well within the window given by NBS, 714 ± 28 μg Pb/g, demonstrating efficient extraction. However, the third moment, estimated by

$$\mu_3 = \sum_{i=1}^{n} \frac{(X_i - \mu)^3}{n}$$

was unusually large and negative indicating that the data was skewed where more low values were obtained than could be expected if the data were normally distributed. The third moment is a highly sensitive parameter and can be strongly affected by a few low values even with a data set this large ($n = 148$).

Data were reviewed to identify the possible cause of the bimodal distribution. Data subsets were generated by aliquot weight and discrete bottle derivation. Frequency distributions of pertinent subsets are presented in Figures 5-12 and 5-13. 1-g and 5-g aliquot data were compared statistically to define the affects of the noted precipitation in the 5-g extracts. The results are summarized in Table 5-2.

TABLE 5-2. COMPARISON OF NBS SRM 1645, RIVER SEDIMENT DATA

	n	μ_3	$\sqrt{B_1}$	$\bar{X}$	1s	2s	3s	$S_{\bar{x}}/\bar{X}$ (%)
Total Data Set	148	-9,846	0.654	726	28	56	84	3.9
5-g Aliquots	70	-11,580	0.558	723	33	66	99	4.6
1-g Aliquots	78	-3,660	0.548	728	23	46	69	3.2

None of these means are statistically different from one another or from the certified value. However, both the lower mean and higher standard deviation of the 5-g aliquot are consistent with precipitation occurring.

FREQUENCY DISTRIBUTION OF DATA FOR NBS SRM 1645,RIVER SEDIMENT (TOTAL PROGRAM)

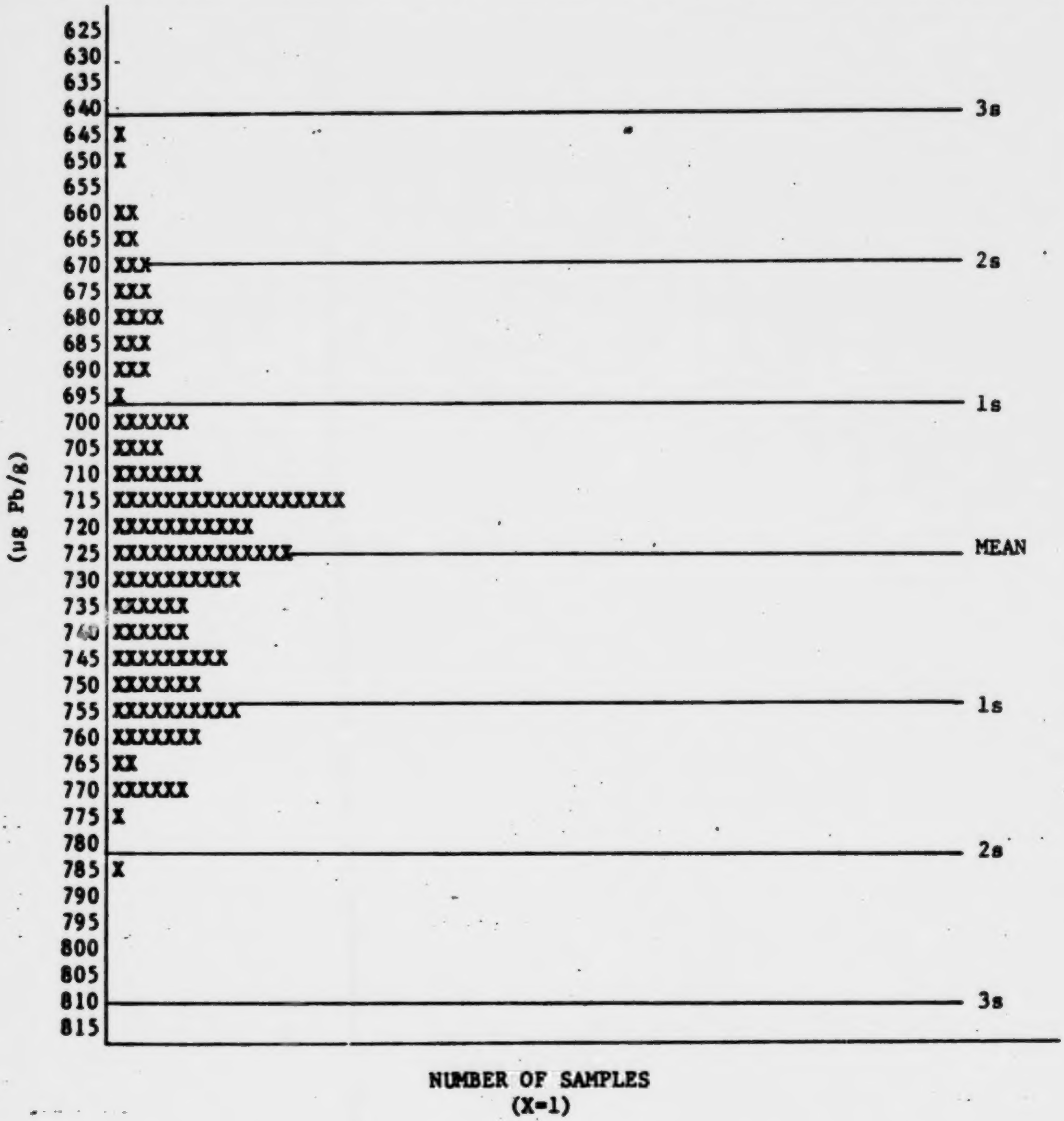

Figure 5-11. Frequency distribution of data for NBS SRM 1645, river sediment (total program).

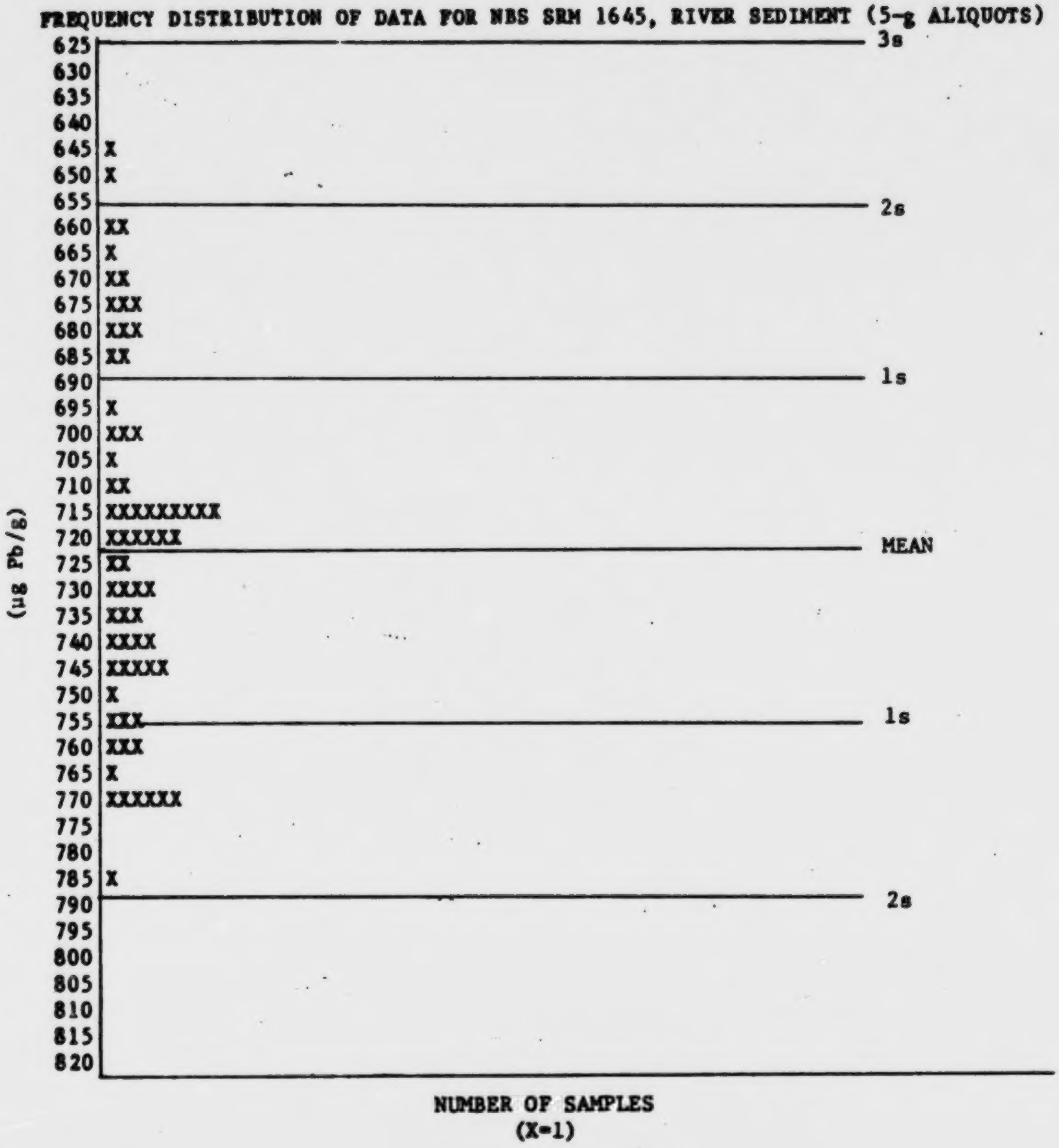

Figure 5-12. Frequency distribution of data for NBS SRM 1645, river sediment (5-g aliquots).

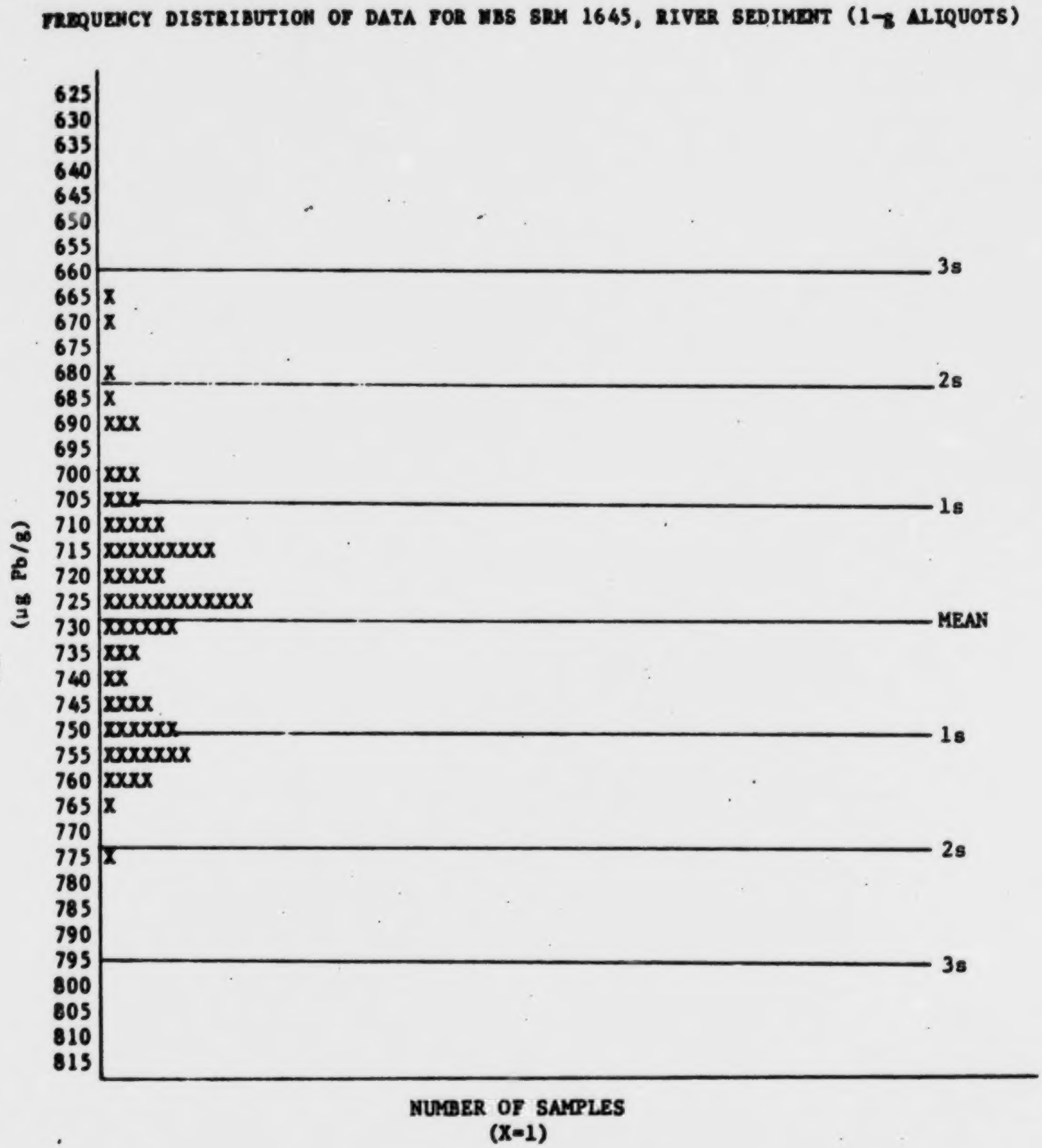

Figure 5-13. Frequency distribution of data for NBS SRM 1645, river sediment (1-g aliquots).

APPENDIX G

PROTOCOL FOR USE OF X-RAY FLUORESCENCE LEAD IN PAINT ANALYZER

PROTOCOL FOR USE OF X-RAY FLUORESCENCE LEAD IN PAINT ANALYZER

I What Is An XRF and How Does It Work

II How To Use An XRF In Field Study

III Glossary Of Terms

I What Is An XRF

An X-ray Fluorescence Lead in Paint Analyzer (XRF) is a small portable instrument useful for making lead determinations in dried wall paint in the field. It can make quantitative measurements of the amount of lead in dry coverings such as paint, varnish, laquer, or other coverings over a variety of substrate such as wood, plaster, gypsum board, and other materials.

The principle of operation is X-ray fluorescence. The technique is essentially that used in conventional X-ray analysis, that is characteristic X-rays are excited in the sample by an external source of radiation and measurements of their intensities yield the concentration of the sample. An elment, if exposed to X-rays of a wavelength shorter than the wavelength of its X-ray absorption edge will absorb some of the incident X-rays and emit its own characteristic X-ray. The XRF uses a small radioisotopic source instead of an X-ray machine for the source of excitation and the X-rays that are characteristic of lead are converted into electronic signals and counted in the analyzer.

The radioisotopic source is hermetically sealed, physically quite small, no larger than a dime and has a half-life from 9 months to slightly longer, depending on which brand of XRF is used. When exposed to the low energy radiation in the source, lead may be detected through several layers of nonleaded paint. The instrument response is a function of the total amount of lead within a given cross sectional area of the painted surface, even though the X-rays are actually reading depth as well as length and width. Readouts are given in terms of the total amount of lead per unit area or the amount of lead expressed as milligrams per square centimeter ($mg/cm^2 Pb$).

The properties that enable the instrument to make rapid, nondestructive readings in the field are also reason to treat the machine with respect. The instrument contains a radioactive source which emits ionizing radiation although at a very low level when compared to an X-ray mechanism (about 10^7 X-rays/sec). Even though the instrument has the source well protected, and the source is only exposed when the trigger is depressed, it does represent a potential health hazard through accident, improper use, or unauthorized servicing. Therefore, a word about radiation is in order.

Radiation is attenuated by three things: (1) time, (2) distance, and (3) mass.

1. When the source is new it will be either at 10 or 2.5 millicuries (Cobolt-57). In approximately 270 days the source will have lost about 50 percent of its activity. This means that the source will have to be exposed longer to the sample analyzed to achieve the accuracy available when the source is new. In a practical sense the Princeton Gamma Tech XR-3 may be used for up to 15 months and the Texas Nuclear Pb-3 for 24 months before the counting time becomes excessive and the data unreliable. The characteristics of a radioactive source are such that use of the instrument has no effect on half-life. An XRF on the shelf will deteriorate at the same rate as one in daily use, insofar as the life of the source is concerned.

2. Distance attenuates all radiation. Exposure to the radioactive decay process is lessened by the farther away one is from the source. Since the XRF's are hand held instruments it is not possible to consider anything further away than arms length as a working procedure, nor is it necessary. The photon output is so low that the dose rate in the Pb-3, with a new source is less than one milliroentgen per hour at one foot directly in the beam with the shutter open. On the Pb-3, exposure is comparable and at 100 centimeters it is less than .01 mR/hr., shutter open or closed. Nevertheless, prudence would dictate that the instrument be transported on the back seat or floor of the automobile rather than on one's lap.

3. The greater the mass surrounding the source, the greater the safeguard. Air will provide some shielding; a lead shield will offer the best protection per unit of volume. Lead shielding is neither necessary nor desirable in a hand held instrument. The source capsule is of welded stainless steel construction with a small 0.3 mm stainless steel window. The source material itself is bound in the form of a ceramic enamel and is quite rugged. The radiation is emitted in one direction only - through the "window" mentioned previously and the source holder assembly is constructed with shielding material so that the only significant radiation is emitted in one direction only when the shutter is opened. The shutter opens only when the trigger is pulled or pressed and closes when released. Th shutter itself is a thick tungsten alloy disc which effectively attenuates the low radiation to a safe level.

II How To Use An XRF In A Field Study

An XRF can give a better environmental profile for lead in paint in the living environment than wet chemistry or any other system. It can do this faster and cheaper than other procedures with better acceptance from study participants since it is nondestructive on wall coverings.

What the XRF cannot do is give a reading in terms of percent of lead in the sample tested. The XRF readings have virtually no quantitative percentage relationship to values determined by wet chemistry. It does give a better assessment of the degree of hazard since it measures the total amount of lead in the area measured. An example to illustrate the point: A room is painted 10 times with 1% leaded paint and wet chemistry gives us a reading of 1% lead in the paint sample even though the amount of lead present was increased with each painting. An XRF would detect the total amount of lead present. The problem is compounded when nonleaded paint is applied over leaded paint and assayed by wet chemistry for then the percent of lead goes down in relationship to the sample. This has little effect on the XRF.

For these reasons the CDC has recommended that hazardous paint be determined by XRF. Any reading on an XRF ≥ 0.7 mg/cm^2 is considered positive. Percent is most useful when used to determine lead as a fraction of the non-volatile liquid paint in the can.

All XRF's should be checked daily for accuracy. The only practial way to do this is by use of the lead standards made by the National Bureau of Standards for the Department of Housing and Urban Development and supplied with each instrument. These lead foil standards allow the user to check the XRF for accuracy before leaving for the field and during the day. It is good practice to check the instrument before and after each dwelling screened. Following this practice there is never a reason to complete a project and then question the data on the basis of unreliability of XRF readings.

The decay of a radioactive source is a random process and each measurement is subject to statistical uncertainty. The XRF will rarely give identical readings on the same surface. The difference should not be greater than 0.5 mg/cm^2 and good procedure in the field is to take three readings and record the middle of the three (not necessarily the second of the three). This gives a reasonable balance between accuracy and time required for the determination. One reading should never be taken by itself.

The XRF should be utilized in the same manner in each dwelling in order that the data generated can be aggregated and houses and apartments compared. The Environmental Lead Survey form should be completed on all participants and rechecked before leaving the premises. Data from the instrument is obtained by taking three readings at each "station" and a single figure recorded. One station per room will be adequate for screening the dwelling. The readings should be taken in the area where the child plays and where the child can reach, approximately one foot from the floor. Readings will generally begin where the interview takes place and proceed throughout the house, making certain that the rooms where the child spends time are checked.

Readings should be taken on the front entry door, porch, etc. and the rear. Fences, garages, and utility buildings should also be checked. In a five-room house there will usually be approximately 7-8 stations where readings are taken, one entry, one exit, one in each room plus the bathroom. This should take less than half an hour with a relatively new source in the instrument.

Instructions for Completion of Environmental Lead Survey Form

Site Surveyed	Record the address of the premises surveyed
Date	Record the date of the survey
By	Record the name of the person making the survey (team number may suffice)
Phone	Record the number where the parent may be contacted
Instrument Number	Record the serial number of the XRF instrument used in the survey
XRF Standard	Record the value of the lead standard used to check XRF calibration
XRF Reading	Record the XRF reading on the standard at beginning of survey
Key	Record X on appropriate wall, record the actual XRF reading if above 0.7 mg/cm^2 under "comments"
Wall	Indicate the location of "lead hazard" on wall of each room by marking an X in the appropriate triangle. Lead source is any XRF reading in excess of 0.7 mg/cm^2
Condition/Chip-Peel	Record any state of disrepair on the wall in each room by making an X in the appropriate box
Conditions/Trim	Record the location of a lead source on the trim of each room by marking an X in appropriate triangle
C	Indicate a lead source on ceiling of any room by marking an X
F	Indicate a lead source on floor of each room by marking an X
Conditions/Chip-Peel	Indicate disrepair of ceiling or floor of each room by marking an X in the appropriate box
XRF-End of Survey	Record the XRF reading of the lead standard at time inspection ended
Sketch	Diagram of dwelling. Draw in pencil, be as accurate as possible. Complete on site. Show house in relation to street, other houses, etc. Show location where dust sample was obtained. Show location of soil sampling areas. Two story houses should have separate drawings for each floor.

Glossary of XRF Related Terms

Accuracy	Accuracy is defined as the plus or minus value within which 66% of readings will differ from the average for at least 10 repeated measurements on a lead foil standard placed on any variety of lead free substrates.
Precision	Here it means the plus or minus amount within which the reading represents the processed information from the measuring head.
Lower Limit of Detection	The lowest reading for which confidence is greater than 85% that the lead concentration is not zero. The lower limit of detection may be reduced by averaging repeated measurements.
Radioactive Source	The radioactive excitation source hermeticaly sealed in the XRF containing cobolt-57 of either 10 or 2.5 millicuries maximum strength.
Repeatibility	The plus or minus difference from the average value of many repeated measurements within which 66% of the individual measured values will fall. Other values of the repeatibility can be obtained by alteration of the measuring time interval.
Standardization	Adjustment in XRF to adjust time source is exposed to test specimen to account for weakening of radiation beam of source decay.
Gain Control	Used to compensate for changes in accuracy and precision due to aging and drift of electronic components.
Substrate Absorption Effects	A change in the reading on the instrument and __________ to material either over or beneath the lead having a relatively high mass absorption coefficient at the existing of fluorescening energy level.
Analysis Time	The time the shutter is open and the test specimen is being irradiated by the radioactive source. Analysis time is increased as the source ages.
Calibration	Adjustment of the XRF to give readings in agreement with those on a standard reference material which have been obtained by a standard reference method. This may be accomplished either manually or automatically depending on the brand of instrument under consideration.

Negative Reading	A reading below 0.0 on the machine can occur when the calibration curve plotted in the instrument intercepts the count axis at some small positive or negative value when the concentration is near zero.
Power Pack	Rechargeable nickel-cadmium batteries used to power the electronics in the lead analyzer. The XRF's have built in battery chargers that should be charged each night.
Station	A spot on the wall or other painted surface where the XRF is placed in order to take the three readings required for the record.

Environmental Lead Survey

Site Inspected ______________________ Date __________ By ______________
Owner ______________________________ Phone __________
Address ______________________________________
City _______________________________ Zip __________ ______________
Instrument No. ____________________ XRF Standard _____ XRF Reading __________ (Start)

KEY ☒ •Represents wall nearest the street: X - Indicates area with Lead paint surfaces in disrepair; C - Indicates ceiling; F - Indicates floor.

Area	Wall	Condition		Trim Work	Condition		C	F	Condition		Comments
		Chip	Peel		Chip	Peel			Chip	Peel	
Living R.	☒			☒							
1) Bed R.	☒			☒							
2) Bed R.	☒			☒							
3) Bed R.	☒			☒							
Bathroom	☒			☒							
Kitchen	☒			☒							
Dining R.	☒			☒							
Hallway	☒			☒							
Other	☒			☒							
Exterior	☒			☒							

Sketch not drawn to scale to illustrate Locations Only

XRF Reading at end of __________
Inspection ______________

APPENDIX H

GCA'S QUALITY ASSURANCE PROJECT PLAN AND WORK PLAN

DALLAS LEAD STUDY
DA 68-02-3168

July 22, 1982

GCA QUALITY ASSURANCE PROJECT PLAN

APPENDIX TO EMSL-LV
QUALITY ASSURANCE PROJECT PLAN
DALLAS LEAD STUDY
DA 68-02-3168

EPA CONTRACT NO. 68-02-3168
(GCA 1-619-080)

APPROVAL:

GCA Project Manager Kenneth D McGeyer Date 7/22/82
GCA QA Manager Rose Mary Ellersick Date 7/22/82
EPA Project Officer ______________________ Date __________
EPA QA Manager ______________________ Date __________

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1.0 PROJECT DESCRIPTION AND ORGANIZATION

1.1 PROJECT DESCRIPTION

Data collected by the city of Dallas and the University of Texas at Arlington, have shown that some soils collected in the immediate vicinity of the Dixie Metal Company (DMC) and the RSR Corporation (RSR) have lead levels that exceed 1,000 μg/g. In addition, blood samples collected from a number of individuals living in the vicinity of these two secondary lead smelters have contained lead levels that exceed 30 μg/dl.

The U.S. Environmental Protection Agency (EPA), the U.S. Public Health Service's Center for Disease Control (CDC) and the City of Dallas have designed an environmental multimedia monitoring study that includes the analysis of soils, house dust, paint and blood samples collected near these two smelter sites. As two major sources of lead are suspected, one from the smelters and one from automobile emissions, a third geographical area identified as a Reference Area (REF) has also been selected for sampling. The selection of a REF site will permit an assessment of the smelter lead contribution, as the REF site will contain as nearly as possible all physical and biological parameters exhibited at the RSR and DMC sites except for the smelter emissions.

The data obtained from this monitoring effort will be used to correlate blood lead levels with selected environmental media. Also, the data will be used to identify geographical areas that may require a remedial response.

This program will include collection of blood samples from pre-school aged children as well as collection of house dust samples and the measurement of lead in paint from the homes in which these children reside. In addition, soils located in the immediate vicinity of homes, at parks, playgrounds, schools and other designated sampling grid locations will be collected.

GCA/Technology Division is currently under contract to EPA to manage and staff the Sample Bank for this program in Dallas, Texas and analyze approximately 5000 soil samples for lead according to the analytical procedures provided by EPA. This Quality Assurance Project Plan will detail the procedures which will be implemented to ensure the validity of all data generated during this program. Appendix B to this QA Plan contains the Analytical Procedures and Analytical Equipment Maintenance and Troubleshooting Procedures.

This QA Plan is prepared as an appendix to the Quality Assurance Plan for the Dallas Lead Study prepared by the EPA-Environmental Monitoring Systems Laboratory, Las Vegas (EMSL-LV). This Plan follows the outline suggested for contractor's QA Plans in the EMSL-LV QA Plan rather than the 16 point format defined in QAMS 005/80. The EMSL-LV Plan should be consulted for more detail as noted herein.

1.2 PROJECT ORGANIZATION

Figure 1-1 presents GCA's organization chart for this project showing the individuals responsible for each element of the overall task. The key individual responsible for QA will be Ms. RoseMary Ellersick, the Division QA Manager, who reports directly to the Division General Manager. Her responsibilities will include the following:

- Review and approve the QA Project Plan.
- Perform audits of recordkeeping and documentation, quality control program and analytical performance.
- Initiate or follow up on corrective action procedures.
- Prepare QA/QC section of Final Report.

The laboratory QC Coordinator, Ms. Mary Kozik, will oversee and implement the ongoing analytical QC program, she will be assisted in this task by Ms. Barbara Myatt. Their responsibilities will include:

- Maintaining control charts on instrument calibration, laboratory control samples and "blind" QC samples.
- Preparing and inserting "blind" QC samples into the project sample load.

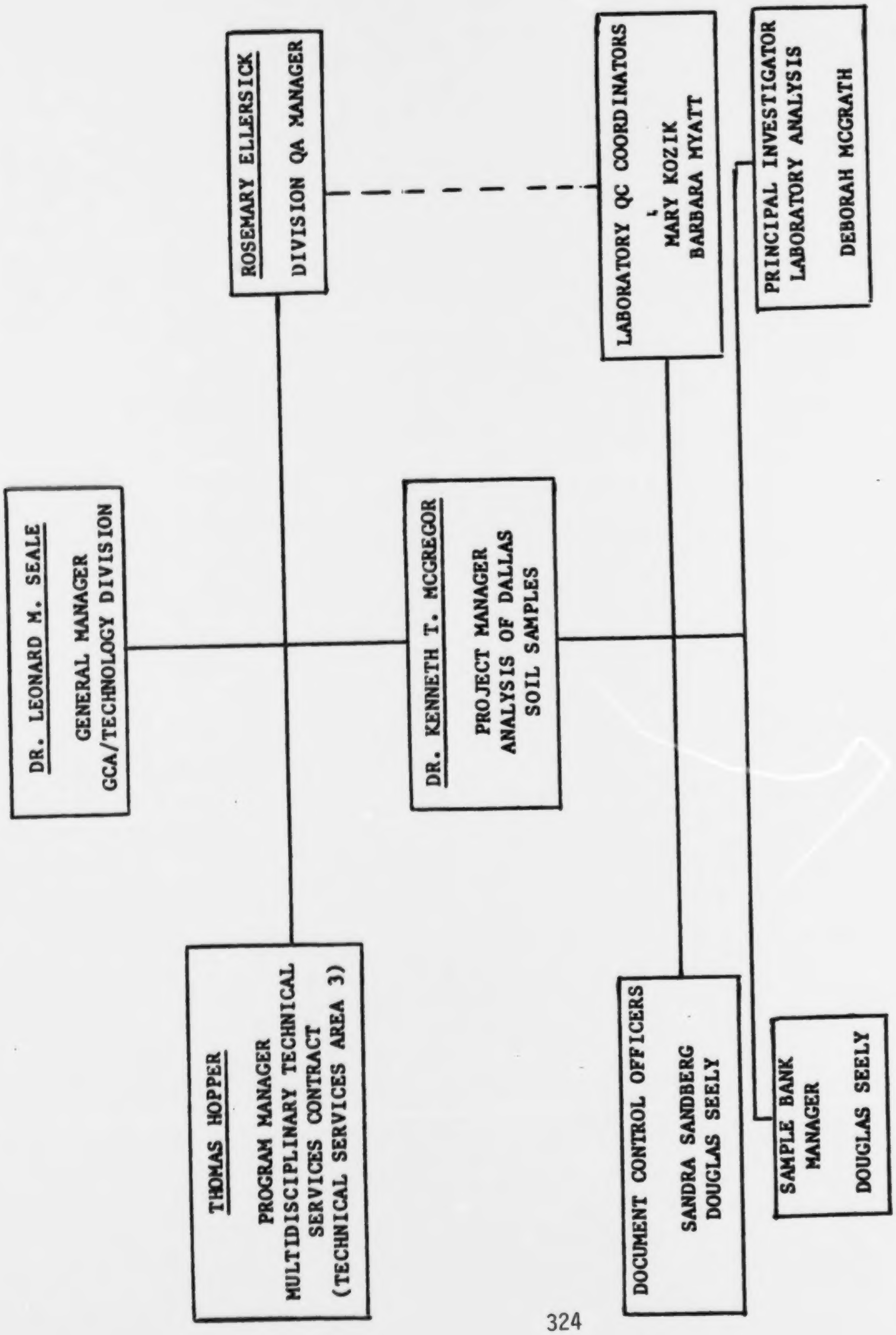
DR. LEONARD M. SEALE
GENERAL MANAGER
GCA/TECHNOLOGY DIVISION
THOMAS HOPPER
PROGRAM MANAGER
MULTIDISCIPLINARY TECHNICAL
SERVICES CONTRACT
(TECHNICAL SERVICES AREA 3)
DR. KENNETH T. MCGREGOR
PROJECT MANAGER
ANALYSIS OF DALLAS
SOIL SAMPLES
ROSEMARY ELLERSICK
DIVISION QA MANAGER
DOCUMENT CONTROL OFFICERS
SANDRA SANDBERG
DOUGLAS SEELY
LABORATORY QC COORDINATORS
MARY KOZIK
BARBARA MYATT
SAMPLE BANK
MANAGER
DOUGLAS SEELY
PRINCIPAL INVESTIGATOR
LABORATORY ANALYSIS
DEBORAH MCGRATH

Figure 1-1. Program organization and responsibility.

- Reviewing all routine laboratory QC results.
- Initiating corrective actions where necessary.
- Assisting in preparation of QA/QC section of final report.

2.0 MAJOR PROJECT ELEMENTS

2.1 SAMPLE BANK

GCA/Technology Division will manage and staff a Sample Bank operation in Dallas, Texas. This will include responsibility for filing, storing, and dispensing sampling equipment and all custody documents such as chain of custody forms and sample collection and analytical tags.

Quality control samples, such as performance evaluation samples will be received at the sample bank from EPA or other preparing laboratories and inserted in the sample group to be shipped to the analytical laboratories.

Collected samples will be accepted at the sample bank and logged into a master log. Since each sample will be assigned a new sample bank number and sample tag, it will be possible to submit duplicate and spiked samples to analytical laboratories as blind QC samples, indistinguishable from collected samples. Sample bank operating procedures are fully described in Attachment 2 to EPA's Dallas Lead Investigation Soil Sampling Protocol.

Sample bank personnel will arrange prompt shipment, usually by Federal Express, to the appropriate analytical laboratories. They will notify the laboratory as to the expected date and delivery time of each shipment.

2.2 SAMPLE ANALYSIS

GCA/Technology Division will analyze approximately 5000 soil samples for elemental lead. These analyses will be performed by flame Atomic Absorption Spectrophotometry (AAS) in accordance with the analytical protocol provided by EPA and shown in Appendix B of this QA Plan.

GCA will adhere to all quality assurance/quality control (QA/QC) procedures specified in the analytical procedure or required by the EPA Project Officer.

GCA will analyze up to 700 samples per week and forward results not later than 21 calendar days after sample receipt. In addition, a project file documenting all work performed under this contract will be maintained; if required, copies of all information contained in this file will be provided to the Project Officer within 90 days after acceptance of the report by EPA.

3.0 QA OBJECTIVES

The objective of this program is to provide accurate and precise analysis of soil samples collected in the monitoring area and report the analytical results in a timely manner using standard, comparable formats.

3.1 PRECISION AND ACCURACY

The analytical protocol (Appendix B) contains detailed descriptions of the quality control measures to be employed during this study. The QA objective is to achieve an analytical precision of ±10 percent with an accuracy of 90-110 percent, expressed as percent recovery of lead. The analytical precision for split samples is expected to be ±20 percent. The overall precision and accuracy (sampling, sample preparation and analysis) are expected to be ±20 percent and 80-120 percent, respectively. Control charts will be maintained to monitor precision and accuracy throughout the program, actual precision and accuracy achieved will be reported.

3.2 COMPARABILITY

The following procedures will be followed to ensure the comparability of the analytical data resulting from this study:

- Use of standard written analytical procedures.
- Use of standard analytical data forms.
- Use of standard reporting units specified by EPA.

4.0 DOCUMENT CONTROL PROCEDURES

The purpose of document control is to ensure that all project documents will be accounted for when the project is complete. The procedures outlined in the Dallas Lead Study QA Project Plan, Section 4.0 will be followed and the numbering systems provided there and in Attachment 2 to the Dallas Lead Investigation Soil Sampling Protocol will be used. GCA's QA Manager, RoseMary Ellersick, will oversee the implementation of appropriate procedures by the Field and Laboratory Document Control Officers.

4.1 FIELD DOCUMENT CONTROL

The onsite Sample Bank Manager, Douglas Seely, will serve as Field Document Control Officer (DCO) for all controlled documents pertaining to sample collection and identification, custody, preparation, and shipment to analytical laboratories. He will oversee the distribution and documentation of the appropriate forms to the sampling teams, and the maintenance of the Sample Bank Master Log for this project. He will ensure that sample collection tags and custody forms are completely and legibly filled out before custody of the corresponding samples is transferred to the Sample Bank.

4.2 LABORATORY DOCUMENT CONTROL

The Laboratory DCO for this program will be Ms. Sandra Sandberg; she will be responsible for issuing, controlling and maintaining records of controlled documents pertaining to analytical work. The numbering systems provided in the QA Plan and Sampling Protocol will be followed with this exception: internal GCA laboratory documents will be identified by the project code 1-619-080.

This follows GCA's routine practice for this contract and has the advantages that it uniquely identifies documents for this work assignment and it is an established procedure, familiar to staff members. The laboratory documents so identified will include internal custody records (pictured in Section 5.0 of this Plan), analysts' notebooks, instrument logbook pages, internal memos, etc.

GCA/Technology Division Analytical Laboratory personnel use bound laboratory notebooks which contain a yellow carbon copy sheet for each page. The analyst is required to note the Project Number and notebook number on each page of the notebook; at the conclusion of the project, the yellow carbon pages will be removed and made a permanent part of the project file. All laboratory notebooks will be assigned by and returned to the GCA Laboratory Document Control Officer.

Analysis Data Reporting Forms and computer printouts will use the DA 68-02-3168 Project Code and no confusion over sample identity should result from the internal use of GCA's Project Number Code.

4.3 OVERALL PROJECT DOCUMENT INVENTORY

At the conclusion of field sampling activity, the Field DCO will assemble and provide an inventory listing of the controlled documents under his responsibility. The Laboratory DCO will follow the same procedure and these inventory listings will form a Table of Contents for the complete project document inventory. The QA Manager will oversee this compilation, coordinating the contents listing if necessary. Table 4-1 lists the expected types of documents to be included. At the conclusion of analytical activity, all controlled documents will be transmitted to EPA.

TABLE 4-1. DOCUMENT CONTROL PROCEDURES

Item	Issued by	Numbering	Interim responsibility	Final responsibility
Sample Collection Tags	Sample Bank	Preserialized	GCA Sample Bank	GCA Field DCO
Site Description Forms	Sample Bank	To be determined	GCA Sample Bank	GCA Field DCO
Analytical Sample Tags	Sample Bank	Preserialized	GCA Sample Bank	GCA Field DCO
Custody Records	Sample Bank	Preserialized	See Section 5.0	GCA Laboratory DCO
Laboratory Notebooks	GCA Laboratory DCO	1-619-080 - Notebook No. Page No.	Analyst	GCA Laboratory DCO
Written Communications	Various People	1-619-080	Recipient	GCA Laboratory DCO
Analytical Data Sheets	Sample Bank	DA-68-02-3168	Analyst	GCA Laboratory DCO

5.0 SAMPLE CUSTODY

The purpose of chain-of-custody procedures is to document the identity of the sample and its handling, from its first existence as a sample until analysis and data reduction are completed and, if required, information derived from it is introduced as evidence in legal proceedings.

5.1 FIELD SAMPLE CUSTODY PROCEDURES

The onsite Sample Bank will dispense the sample containers, sample identification tags, chain-of-custody forms and site identification forms defined in the Dallas Lead Study QA Project Plan, Section 5.0, Revision 1. Sampling equipment may also be included, and the procedures detailed in both the cited QA Plan and Attachment 2 to the Dallas Lead Investigation Soil Sampling Protocol will be followed to ensure that chain-of-custody requirements are met.

GCA's Sample Bank Manager, Douglas Seely, will oversee the operation of these chain-of-custody procedures.

5.2 LABORATORY CUSTODY PROCEDURES

All samples submitted to the GCA/Technology Division Analytical Laboratory will be brought to the Sample Bank Manager, Sandra Sandberg, who will continue the chain of custody by assigning a GCA Control Number to each sample on receipt; this number identifies the sample through all further handling. The sample will be recorded in the bound Master Sample Log under its GCA Control Number. A Master Log page is not depicted here because the hand-written records do not reproduce well; however, each page of the Master Log has the following format:

- GCA Control Number
- Sample Description
- Sample Condition

- Signature of person completing sample record
- Date of Sample Receipt

GCA/Technology Division maintains large, locked, refrigerated and nonrefrigerated storage areas with provision for hazardous material storage. After necessary preservation or subdivision, the Sample Bank Manager will store each sample in the unrefrigerated area under its GCA Control Number.

The Sample Bank Manager will initiate a page (Figure 5-1) for each sample in the Custody Book and ensures that each handling of the sample is appropriately documented. Each analyst working with the sample will first go to the Sample Bank Manager and record in the Custody Book actions taken on the sample thereby maintaining the chain of custody of the original sample.

When sample preparation and analysis procedures necessitate the transfer of samples between two analysts within the laboratory, A Sample Custody Transfer form (Figure 5-2) is required. This document serves as a supplement to the Custody Notebook record of sample handling and becomes part of the permanent project file.

If analytical work on a set of samples cannot be completed in a single day, the sample set will be returned to the Sample Bank for secure storage until analysis resumes.

PROJECT ____________________ GCA CONTROL NO. ______________

LABORATORY ANALYSIS

Sample ID ____

Sample Description ______________________________________

Date Received ____________________ Received By ____________

Procedure	Amount	Name	Date	Comments

Special Instructions/Other Comments ______________________________

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Figure 5-1. Custody book page.

SAMPLE CUSTODY TRANSFER

PURPOSE

Procedure/Analysis required ______________________________
____
____
(General information only--analyst MUST refer to Project File for specific details.)
Instrumentation required ______________________________
____

BACKGROUND

Client ____
Contract (Charge) No. ____________________ Work Order No. ______________

SAMPLES

General description of sample type(s) ______________________________
____
List of samples (by GCA Control No.):
____
____
____
____
Additional samples (QC-blanks, QC-spikes, etc.):
____
____
Total number of samples ______________________________
Comments ____
____
____

TRANSFER

From ______________________________ Date ______________
Received by ______________________________ Date ______________
(Location of samples) ______________________________________)

When completed, make 3 copies--one each for originator, receipient, and Task Manager.
RETURN ORIGINAL TO PROJECT FILE

Figure 5-2. Sample custody transfer.

6.0 ANALYTICAL INTERNAL QC CHECKS

This section describes the quality control procedures which will be used during the analysis of soil samples from the Dallas, Texas study area for lead content. The purpose of the quality control procedures is to ensure the quality of the data and document the precision and accuracy of the procedures used.

6.1 ANALYTICAL METHOD QUALITY CONTROL

Quality control checks on the analytical procedures will include the analysis of laboratory control samples (NBS-SRM 1645, River Sediment), method blanks, spiked samples and extracts, reextraction of residue and use of a procedure for Total Recoverable Lead; the acceptance limits and frequency of each QC procedure are summarized in Table 6-1. The frequency of several QC procedures may be reduced as a result of the preliminary study and/or early analytical results which show the analytical system to be in control.

6.2 QA AUDITS

GCA/Technology Division's quality assurance program includes both performance and system audits as independent checks of the quality of data obtained from sampling, analysis, and data gathering activities. Every effort is made to have the audit assess the measurement process in normal operation. Either type of audit may show the need for corrective action.

The analysis, and data handling segments of a project are checked in performance audits. A different operator/analyst performs these audit operations to ensure the independence of the quantitative results.

In this program, EPA Quality Control Trace Metals concentrates and NBS Standard Reference Material 1645, River Sediment, will be used to assess the analytical work. The laboratory QC Coordinator will direct the inclusion in the sample load of QC samples appropriate to the analyses performed so that they are not recognizable to the analyst. In addition, any appropriate interlaboratory study samples which are available during this program will be analyzed to further audit the analytical work.

TABLE 6-1. SOIL ANALYSIS--INTERNAL QUALITY CONTROL CHECKS

Operation	Control limits	Planned corrective action
1. Calibrate and Standardize AAS daily; analyze EPA Trace Metals Concentrate (WP475 series) after calibration and after every 20 samples.	1. Maintain control chart showing 99 and 95% confidence limits. Acceptable range is 99% confidence interval or 95-105% whichever is less.	1a. Prepare fresh working standards; recalibrate instrument. 1b. Analyze a second EPA Trace Metals concentrate. 1c. Invalidate results, repeat analysis.
2. Method (Reagent) Blank—Three per shift day.	2. No significant concentrations found ($\leq$0.25 μg/ml)	2a. Check reagents and glassware for contamination. 2b. Correct for blank. 2c. Contact EMSL-LV.
3. Spiked Extract--Spike one of every 20 samples with lead nitrate standard at a level equal to that found in the sample or 10 times the instrument detection limit.	3. 90-110% recovery of lead spike.	3a. Repeat on two additional sample extracts. 3b. If either or both of the additional samples are outside control limits, analyze samples by the method of standard additions.
4. Residue Reextraction--For every 20 soil samples extracted, reextract and analyze one sample residue.	4. Calculate percent extraction (first extraction). The initial limit will be 90%; this may be changed after analysis of the first 200 to 400 samples.	4a. Check analytical procedure. 4b. Analyze laboratory control standard. 4c. Invalidate results.
5. Total Recoverable Analysis--Extract one sample out of every 20 samples using the "Total Recoverable" procedure contained in the analytical protocol.	5. Results of Total Recoverable and extraction method should agree within 10%; this may be changed after analysis of the first 200 to 400 samples.	5a. Check analytical procedure. 5b. Analyze a second sample using "Total Recoverable" method. 5c. Invalidate Results.
6. Laboratory Control Sample--Analyze one 5 g sample of NBS-SRM 1645, River Sediment for every 20 samples. If the first 10 results meet the control limits, LC frequency may be reduced to three per shift (3 per 100-150 samples).	6. Report LCS result as percent true value. Prepare control chart and calculate 99 and 95% confidence limits. Acceptable recovery is 90-110% or the 99% confidence interval, whichever is less.	6a. Check analytical procedure. 6b. Analyze second LCS sample. 6c. Invalidate results.

(continued)

TABLE 6-1 (continued)

Operation	Control limits	Planned corrective action
7. Split Extract Analysis—Analyze a second aliquot of one of every 20 soil sample extracts.	7. A precision of 3% is expected; precision is not to exceed 5%.	7a. Recalibrate as in 1. 7b. Analyze a second sample extract in duplicate. 7c. If precision is not within 5%, notify EMSL-LV immediately.
8. Spiked Sample Analysis--For every 20 field samples, spike a duplicate portion of one sample with lead at a level equal to the level found in the sample or at least 10 times the detection limit.	8. Calculate percent recovery; maintain control charts showing percent recovery and develop 99 and 95% confidence limits. Acceptable range for lead recovery is 90-110% or the 99% confidence interval. Acceptable range will be reviewed after analysis of 200 samples.	8a. Check analytical procedure. 8b. Repeat spiked sample analysis on an additional sample. 8c. Contact EMSL-LV for assistance.
9. Blind QC samples--Insert one sample of NBS 1645 River Sediment with each set (100-150) of samples processed.	9. Report blind QC result as percent true value. Acceptable recovery is 90-110% or the 99% confidence limits from LCS analysis.	9a. Check analytical procedure. 9b. Invalidate results.

A system audit will be conducted by the Division QA Manager at least once during the program to ensure that the elements outlined in the Project QA Plan are functioning.

GCA will cooperate fully in any performance or system audits conducted or arranged by EPA.

7.0 DATA REPORTING MANAGEMENT SYSTEM

7.1 DATA REDUCTION AND VALIDATION

Analysis results will be reduced to the specified concentration units using the equations given in the analytical procedure supplied by EPA.

Data validation is the process of filtering data and accepting or rejecting it on the basis of sound criteria. GCA/Technology Division supervisory and QC personnel will use validation methods and criteria appropriate to the analysis. Records of all data will be maintained, even that judged to be an outlying or spurious value. The persons validating the data will have sufficient knowledge of the technical work to identify questionable values.

Analytical data validation procedures will be based upon the results of internal QC checks stated in Section 6.0 of this QA Plan and the precision and accuracy goals outlined in Section 3.0. Figure 7-1 depicts the data reporting scheme.

All calculations will be verified by the Principal Investigator for this task. The data will be reported on coding forms supplied by EPA; all forms will be checked for transcription errors prior to release of the data.

7.2 IDENTIFICATION AND TREATMENT OF OUTLIERS

Any data point which deviates markedly from others in its set of measurements will be investigated; however, the suspected outlier will be recorded and retained in the data set while it is investigated. One or both of the following tests will be used to identify outliers.

Dixon's test for extreme observations[1,2] is an easily computed procedure for determining whether a single very large or very small value is consistent with the remaining data. The one-tailed t test for difference[1] may also be used in this case. Reference 1 contains calculation formats and tables of critical values for these tests. It should be noted that these tests are designed for testing a single value. If more than one outlier is suspected in the same set of data, the statistical sources listed will be consulted and the most appropriate test of hypothesis will be used.

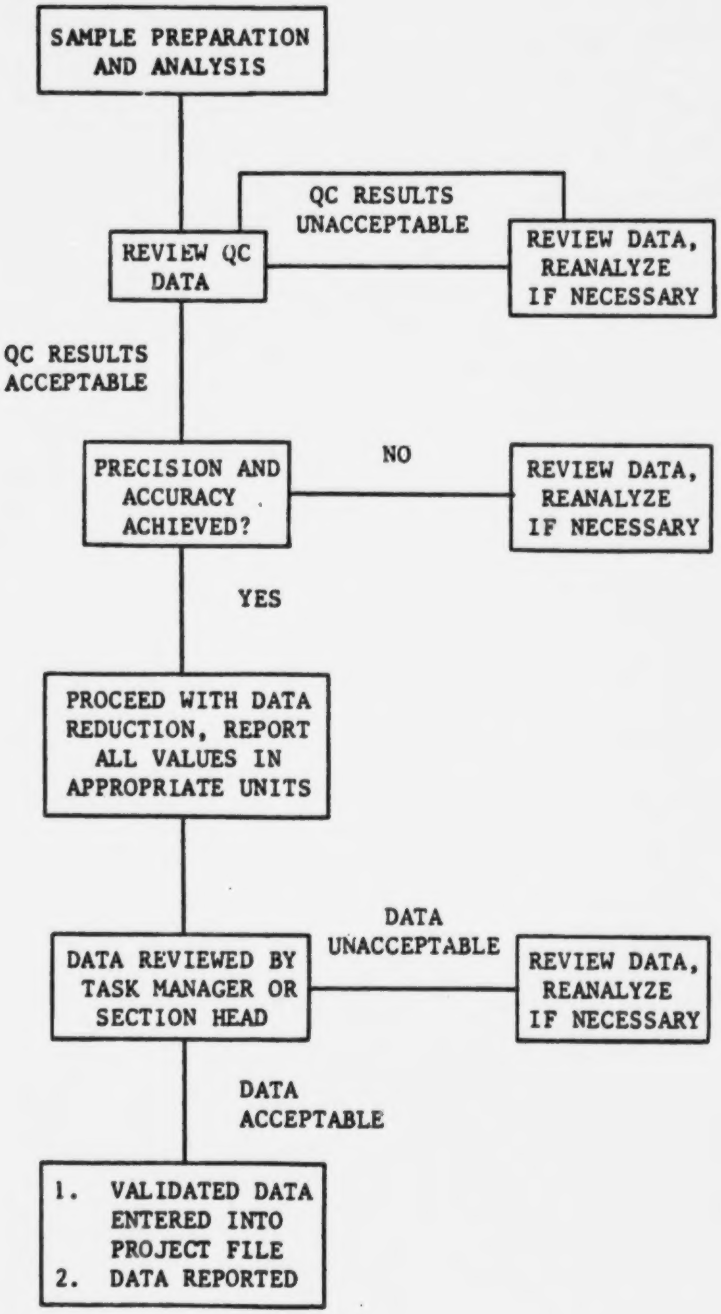

SAMPLE PREPARATION AND ANALYSIS
REVIEW QC DATA
QC RESULTS UNACCEPTABLE
REVIEW DATA, REANALYZE IF NECESSARY
QC RESULTS ACCEPTABLE
PRECISION AND ACCURACY ACHIEVED?
NO
REVIEW DATA, REANALYZE IF NECESSARY
YES
PROCEED WITH DATA REDUCTION, REPORT ALL VALUES IN APPROPRIATE UNITS
DATA REVIEWED BY TASK MANAGER OR SECTION HEAD
DATA UNACCEPTABLE
REVIEW DATA, REANALYZE IF NECESSARY
DATA ACCEPTABLE
1. VALIDATED DATA ENTERED INTO PROJECT FILE
2. DATA REPORTED

Figure 7-1. GCA analytical data reporting scheme.

Since an outlier may result from unique circumstances at the time of sample analysis or data collection, those persons involved in the analysis and data reduction will be consulted. This may provide an experimental reason for the outlier. Further statistical analyses will be performed with and without the outlier to determine its effect on the conclusions. In many cases, two data sets will be reported, one including and one excluding the outlier.

In summary, every effort will be made to include the outlying value in the reported data. If the value is rejected, it will be identified as an outlier, reported with its data set and its omission noted.

8.0 CORRECTIVE ACTION

The acceptance limits for the lead analyses to be conducted in this program will be those stated in the analytical method and summarized in Table 6-1. The corrective actions are likely to be immediate in nature and most often will be implemented by the analyst or Project Manager; the corrective action will usually involve recalculation or reanalysis. GCA's ongoing corrective action policy is described here.

8.1 IMMEDIATE CORRECTIVE ACTION

Specific QC procedures and checklists such as Table 6-1, are designed to help analysts detect the need for corrective action. Often the person's experience will be more valuable in alerting the operator to suspicious data or malfunctioning equipment.

If a corrective action can be taken at this point, as part of normal operating procedures, the collection of poor quality data can be avoided. Instrument and equipment malfunctions are amenable to this type of action and GCA's QC procedures include troubleshooting guides and corrective action suggestions. The actions taken should be noted in field or laboratory notebooks but no other formal documentation is required, unless further corrective action is necessary. These on-the-spot corrective actions are an everyday part of the QA/QC system.

If the problem is not solved in this way, more formalized long-term corrective action may be necessary.

8.2 LONG-TERM CORRECTIVE ACTION

The need for this action may be identified by standard QC procedures, control charts, performance or system audits. Any quality problem which cannot be solved by immediate corrective action falls into the long-term category. GCA uses a system to ensure that the condition is reported to a person responsible for correcting it who is part of the closed-loop action and follow-up plan.

The essential steps in the closed-loop corrective action system are:

- Identify and define the problem.
- Assign responsibility for investigating the problem.
- Investigate and determine the cause of the problem.
- Determine a corrective action to eliminate the problem.
- Assign and accept responsibility for implementing the corrective action.
- Establish effectiveness of the corrective action and implement it.
- Verify that the corrective action has eliminated the problem.

Figure 8-1 shows the sequence of activities in the loop which is designed to assure action and follow up on all reported problems. Documentation of the problem is important to the system. A Corrective Action Request Form (shown in Figure 8-2) is filled out by the person finding the quality problem. This form identifies the problem, possible causes and the person responsible for action on the problem. The responsible person may be an analyst, field team leader, department QC coordinator or the QA Manager. If no person is identified as responsible for action, the QA Manager investigates the situation and determines who is responsible in each case.

The Corrective Action Request Form includes a description of the corrective action planned and the date it was taken, and space for follow-up. The QA Manager checks to be sure that initial action has been taken and appears effective and, at an appropriate later date, checks again to see if the problem has been fully solved. The QA Manager receives a copy of all Corrective Action Forms and then enters them in the Corrective Action Log. This permanent record aids the QA Manager in follow-up and makes any quality problems visible to management; the log may also prove valuable in listing a similar problem and its solution.

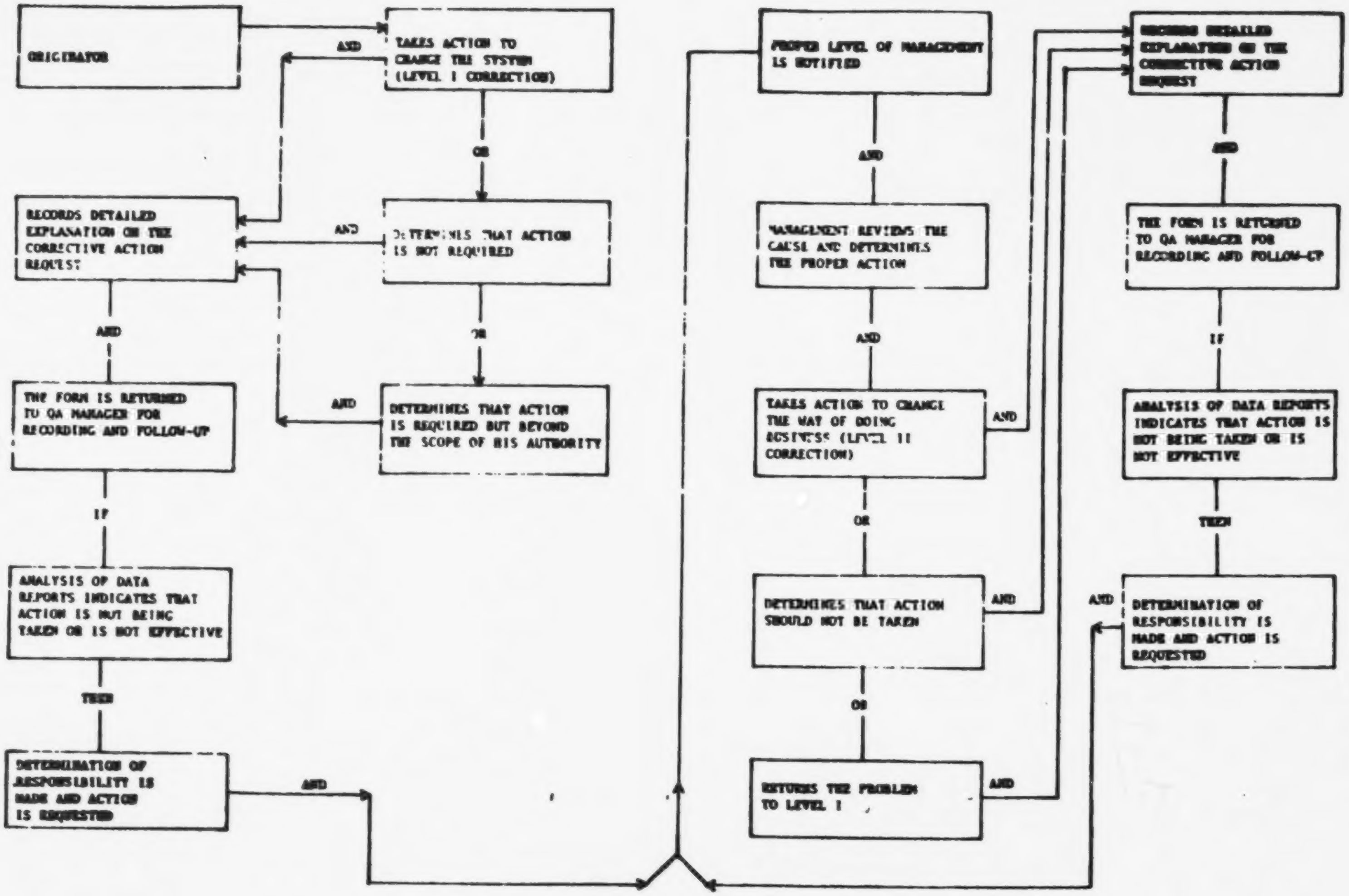

ORIGINATOR
AND
TAKES ACTION TO CHANGE THE SYSTEM (LEVEL I CORRECTION)
OR
DETERMINES THAT ACTION IS NOT REQUIRED
OR
DETERMINES THAT ACTION IS REQUIRED BUT BEYOND THE SCOPE OF HIS AUTHORITY
AND
AND
RECORDS DETAILED EXPLANATION ON THE CORRECTIVE ACTION REQUEST
AND
THE FORM IS RETURNED TO QA MANAGER FOR RECORDING AND FOLLOW-UP
IF
ANALYSIS OF DATA REPORTS INDICATES THAT ACTION IS NOT BEING TAKEN OR IS NOT EFFECTIVE
THEN
DETERMINATION OF RESPONSIBILITY IS MADE AND ACTION IS REQUESTED
AND
PROPER LEVEL OF MANAGEMENT IS NOTIFIED
AND
MANAGEMENT REVIEWS THE CAUSE AND DETERMINES THE PROPER ACTION
AND
TAKES ACTION TO CHANGE THE WAY OF DOING BUSINESS (LEVEL II CORRECTION)
OR
DETERMINES THAT ACTION SHOULD NOT BE TAKEN
OR
RETURNS THE PROBLEM TO LEVEL I
AND
AND
AND
RECORDS DETAILED EXPLANATION ON THE CORRECTIVE ACTION REQUEST
AND
THE FORM IS RETURNED TO QA MANAGER FOR RECORDING AND FOLLOW-UP
IF
ANALYSIS OF DATA REPORTS INDICATES THAT ACTION IS NOT BEING TAKEN OR IS NOT EFFECTIVE
THEN
DETERMINATION OF RESPONSIBILITY IS MADE AND ACTION IS REQUESTED
AND

Figure 8-1. A closed-loop corrective action system.

BEST COPY AVAILABLE
FOR CLARIFICATION CONTACT
KENNETH W. BROWN (702) 798-2214

CORRECTIVE ACTION REQUEST FORM NO. ________

Originator ____________________ Date ____________________

Person Responsible
for Replying ____________________ Contract
Involved ____________________

Description of problem and when identified: ____________________

State cause of problem, if known or suspected: ____________________

Sequence of Corrective Action: (If no responsible person is identified, bring this form directly to QA Manager.)

State Date, Person, and Action Planned:

CA Initially Approved By: ____________________ Date: ____________

Follow-up Dates ____________________

Final CA Approval By: ____________________ Date: ____________

Information copies to:

RESPONSIBLE PERSON: ____________________

QA MANAGER: ____________________

DEPARTMENT MANAGER: ____________________

Figure 8-2. Correction action request form.

9.0 REFERENCES

1. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume 1 - Principles, EPA-600/9-76-005, Research Triangle Park, North Carolina. 1976.

2. Dixon, W. J., Processing Data for Outliers, Biometrics, 9(1):74-89, 1973.

PART 1. ANALYTICAL AND CALIBRATION PROCEDURES

DETERMINATION OF LEAD IN SOIL
DALLAS LEAD SMELTER STUDY
(1-619-080)

SCOPE AND APPLICATION

This procedure has been prepared by EPA for use in the analysis of the lead content of soil samples from the Dallas monitoring area.

SUMMARY OF METHOD

A portion of the sample weighing approximately 5 grams is ground to pass through a 100-mesh sieve. The sieved sample is dried, weighed and extracted with nitric acid. The extract is analyzed for lead content by flame Atomic Absorption Spectrophotometry (AAS).

SAMPLE COLLECTION AND PRESERVATION

Samples are stored in polyethylene containers in the unrefrigerated sample bank.

INTERFERENCES

It is difficult to predict the interferences which may be encountered in this analysis. Interferences may be detected through the use of the method of standard additions as specified in the quality control section of this procedure.

REAGENTS

1. Deionized water (D.I. water)
2. Concentrated nitric acid (HNO_3) J. T. Baker Instra-Analyzed Grade
3. NBS SRM 928, Lead Nitrate ($Pb(NO_3)_2$)
4. NBS SRM 1645 River Sediment
5. EPA Trace Metals QC Samples—WP475 Series
6. Calibration Standards
   a. Prepare the 1,000 g/ml stock solution by dissolving 1.5985 g of NBS-SRM 928, Lead Nitrate, in 200 ml of deionized water contained in a 1 liter volumetric flask. Add 250 ml concentrated HNO_3 (Baker Instra-Analyzed Grade) and dilute to volume with deionized water. Transfer to a clean 1 liter nalgene container, label with date prepared, initials and concentration; enter the required information in the standards logbook.

b. Prepare a 100 μg/ml Pb standard solution by diluting 50 ml of the 1,000 μg/ml Pb stock solution to volume in a 500-ml volumetric flask using 1:4 HNO_3. Prepare this solution fresh daily.

c. Prepare fresh calibration standards daily at concentrations of 0.5, 1.0, 2.0, 5.0, 10.0 and 15.0 μg/ml Pb by pipetting 0.5, 1.0, 2.0, 5.0, 10.0 and 15.0 ml, respectively, of the 100 μg/ml standard into a series of 100 ml volumetric flasks and diluting to volume with 1:4 HNO_3.

APPARATUS

1. Glassware--Use only Class A Borosilicate glassware
   a. Beakers--150 ml graduated Pyrex
   b. Volumetric flasks--50 ml, 100 ml, 500 ml and 1,000 ml capacity
   c. Pipettes--These must be calibrated prior to use
   d. Watch glasses/Speedy vaps
   e. Funnels
   f. Glass stirring rods
2. Hot plates and/or block digestors
3. Drying oven
4. Mettler H80 or HL32 Analytical Balance
5. Filter Paper--Whatman 42, ashless
6. Perkin Elmer 460 or Varian AA-6 Atomic Absorption Spectrophotometer
7. Gases for AAS--Air, Acetylene
8. Mortar and pestle, glass
9. 100 mesh sieve, U.S. standard with nylon screen
10. Nalgene containers

CLEANING PROCEDURE FOR LABORATORY APPARATUS

All laboratory glassware used for extraction, analysis and storage must be cleaned according to the following procedure:

1. Wash in hot water using Alconox.

2. Rinse thoroughly three times with hot tap water to remove all of the detergent.

3. Soak for at least 4 hours in an acid bath containing 1:5 (v/v) nitric acid.

4. Remove from acid bath and rinse three times with deionized water in order to remove all traces of acid.

5. Dry the glassware at 105°C.

6. Store cleaned glassware in closed cabinets to protect from dust and fumes.

CALIBRATION OF ANALYTICAL INSTRUMENTATION

Analytical Balance

1. The Mettler H80 and HL32 analytical balance will be calibrated at the start of the study using Class S weights ranging from 1 to 100 g; this will be repeated every 21 days during the course of the program. In addition, the balance calibration will be routinely checked on a daily basis using a single Class S weight close to the expected sample weight. If the readings do not agree within ±0.2 mg of the stated value, the balance will be recalibrated. The results of all balance checks will be recorded in the balance log book.

Instrument Calibration--Perkin Elmer 460 and Varian AA-6 Atomic Absorption Spectrophotometer

1. After allowing sufficient time for lamp warm up, align lamp and burner head. Record all operating conditions in the instrument log book.

2. Aspirate each of the working standards and record the absorbance.

3. Verify instrument calibration by analysis of an EPA Trace Metals QC Sample. The reported value must be within 5 percent of the expected value.

QUALITY CONTROL REQUIREMENTS

In order to minimize bias, the QC samples are intermixed with the field samples, and the analysis sequence following calibration and analysis of the calibration control sample (b) will be randomly changed. Order the analysis sequence for every set of 20 samples in the manner described below, and then refer to a random number table to determine where to start the analysis sequence.

1. Perform calibration and calibration control samples analysis. After every tenth sample, reanalyze a calibration standard.

2. Order 20 field samples (X_1 to X_{20} and corresponding QC samples as follows: X_1, X_2, X_3, a, X_4, X_5, X_6, e, X_7, X_8, X_9, g_1, X_{10}, X_{11}, X_{12}, f, X_{13}, X_{14}, X_{15}, h, X_{16}, X_{17}, X_{18}, i, X_{19}, X_{20}, g_2, j, . . . continue with b and next sequence where: X_1 . . . X_{20} are samples 1 through 20 and where: a, e, g, f, h, i, j, and b are the QC analyses specified below. [QC samples c and d are not included since they are to be treated as samples.]

   Refer to a random number table to determine where to start the analysis sequence (X_1 or e or etc.).

Extensive quality control procedures are required for this program. The required QC procedures are discussed in detail here; Table 1 summarizes the procedures, their frequency and the required corrective action.

The following quality control procedures must be performed at the stated frequency.

a. One reagent blank (3 per shift--at beginning, middle, and end or maximum 1 per 20 field samples)

b. One calibration control sample (1 per 20)

c. Field and sample bank blanks (1 per 40: field blank; 1 per 40: sample bank blank)

d. One split sample (blind) (1 per 20--other split sent to NEIC)

e. One spiked extract (1 per 20)* where the parent sample is X_6

f. One re-extraction of sample residue (1 per 20)* where the parent sample is X_{12}

g. One "total recoverable" analysis (1 per 10)* where the parent samples are X_8 for g_1 and X_{20} for g_2

h. One laboratory control sample (1 per 20) at start of program; after 10 acceptable data points are available frequency may drop to three per shift.

i. One split extract analysis (1 per 20)* where the parent sample is X_8

j. One spiked sample (1 per 20)* where the parent sample is X_8

Each procedure is discussed in detail below.

---

*The frequency of these analyses may be decreased during the study.

TABLE 1. SOIL ANALYSIS--INTERNAL QUALITY CONTROL CHECKS

Operation	Control limits	Planned corrective action
1. Calibrate and Standardize AAS daily; analyze EPA Trace Metals Concentrate (WP475 series) after calibration and after every 20 samples.	1. Maintain control chart showing 99 and 95% confidence limits. Acceptable range is 99% confidence interval or 95-105% whichever is less.	1a. Prepare fresh working standards; recalibrate instrument. 1b. Analyze a second EPA Trace Metals concentrate. 1c. Invalidate results, repeat analysis.
2. Method (Reagent) Blank--Three per shift day.	2. No significant concentrations found (≤0.25 μg/ml)	2a. Check reagents and glassware for contamination. 2b. Correct for blank. 2c. Contact EMSL-LV.
3. Spiked Extract--Spike one of every 20 samples with lead nitrate standard at a level equal to that found in the sample or 10 times the instrument detection limit.	3. 90-110% recovery of lead spike.	3a. Repeat on two additional sample extracts. 3b. If either or both of the additional samples are outside control limits, analyze samples by the method of standard additions.
4. Residue Reextraction--For every 20 soil samples extracted, reextract and analyze one sample residue.	4. Calculate percent extraction (first extraction). The initial limit will be 90%; this may be changed after analysis of the first 200 to 400 samples.	4a. Check analytical procedure. 4b. Analyze laboratory control standard. 4c. Invalidate results.
5. Total Recoverable Analysis--Extract one sample out of every 20 samples using the "Total Recoverable" procedure contained in the analytical protocol.	5. Results of Total Recoverable and extraction method should agree within 10%; this may be changed after analysis of the first 200 to 400 samples.	5a. Check analytical procedure. 5b. Analyze a second sample using "Total Recoverable" method. 5c. Invalidate Results.
6. Laboratory Control Sample--Analyze one 5 g sample of NBS-SRM 1645, River Sediment for every 20 samples. If the first 10 results meet the control limits, LC frequency may be reduced to three per shift (3 per 100-150 samples).	6. Report LCS result as percent true value. Prepare control chart and calculate 99 and 95% confidence limits. Acceptable recovery is 90-110% or the 99% confidence interval, whichever is less.	6a. Check analytical procedure. 6b. Analyze second LCS sample. 6c. Invalidate results.

(continued)

TABLE 1 (continued)

Operation	Control limits	Planned corrective action
7. Split Extract Analysis--Analyze a second aliquot of one of every 20 soil sample extracts.	7. A precision of 3% is expected; precision is not to exceed 5%.	7a. Recalibrate as in 1. 7b. Analyze a second sample extract in duplicate. 7c. If precision is not within 5%, notify EMSL-LV immediately.
8. Spiked Sample Analysis--For every 20 field samples, spike a duplicate portion of one sample with lead at a level equal to the level found in the sample or at least 10 times the detection limit.	8. Calculate percent recovery; maintain control charts showing percent recovery and develop 99 and 95% confidence limits. Acceptable range for lead recovery is 90-110% or the 99% confidence interval. Acceptable range will be reviewed after analysis of 200 samples.	8a. Check analytical procedure. 8b. Repeat spiked sample analysis on an additional sample. 8c. Contact EMSL-LV for assistance.
9. Blind QC samples--Insert one sample of NBS 1645 River Sediment with each set (100-150) of samples processed.	9. Report blind QC result as percent true value. Acceptable recovery is 90-110% or the 99% confidence limits from LCS analysis.	9a. Check analytical procedure. 9b. Invalidate results.

a. Reagent Blanks--Prepare and analyze three reagent blanks per day (beginning, middle and end of day) or a maximum of one per set of 20 samples on each day that soil extractions are performed. Carry the reagent blank through the digestion procedure with project samples.

Report results on the Soil Sample Quality Assurance Form. If the concentration of lead in the reagent blank exceeds 0.25 μg/ml (one-half the lowest calibration standard) then sources of contamination should be identified and eliminated, and EMSL-LV should be contacted immediately for assistance.

b. Calibration Control Samples--In order to verify the instrument calibration, analyze a known control sample, such as the EPA Quality Assurance Water Samples--Trace Metals--WP475 (prepared by EMSL-CIN), immediately following calibration and after every 20 samples. Dilute the concentrate to fall within the calibration range of the AAS for lead.

Calculate the percent recovery, using the certified lead concentration of the control sample, as diluted, and the analyzed concentration, according to the following equation:

$$\%\ \text{recovery} = A/B \times 100$$

where: A = analyzed concentration of lead

B = certified concentration of lead (as diluted).

Results are to be reported on the Soil Sample Quality Assurance Form.

Maintain control charts on the percent recovery and establish through repetitive analyses the 99 and 95 percent confidence limits (control and warning).

If the percent recovery is outside of the acceptable range of 95 to 105 percent or outside the 99 percent confidence limits, whichever is less, then corrective action such as recalibration and preparation of fresh working and/or stock lead solutions must be taken.

c. Field and Sample Bank Blank Analyses--For every 40 soil samples collected in the field one soil container filled with DI water will be sent to the Sample Bank and then forwarded to the laboratory. For every 40 soil samples processed through the sample bank, DI water will be similarly processed and then forwarded to the laboratory in a soil container. For each blank, the entire contents of the container will be transferred to a beaker, carefully evaporated to near dryness and then processed in the same manner as soil samples. Results are to be reported on the Soil Sample Quality Assurance Form.

d. Split Sample (Blind)--One in 20 soil samples sent to the Sample Bank will be split and forwarded as a blind duplicate to the participating laboratory.

e. Spiked Extract Analysis--For each 20 soil samples, spike one sample extract with lead nitrate to determine the effect of the extract matrix on the accuracy of the analysis procedure. The parent soil sample will be sample X_6.

The predetermined soil sample is extracted and analyzed in the usual manner.

To an aliquot of this sample extract or its final dilution, if applicable, add an amount of lead (as lead nitrate) approximately equal to the lead content determined for this extract or its final dilution, if applicable, but greater than 10 x the instrumental detection limit. This is best accomplished with the 1,000 μg/ml Pb standard stock solution using a calibrated micropipet such that the extract volume is not appreciably changed.

Analyze the spiked extract and calculate the percent recovery of the spike using the following equation:

$$\% \text{ recovery} = (A-B)/C \times 100$$

where: A = analyzed total lead content of the spiked extract

B = analyzed lead content of the unspiked extract

C = lead content of spike.

Report the results on the Soil Sample Quality Assurance Form. If the percent recovery is outside of the acceptable range of 90 to 110 percent, repeat the spiked extract analysis on two additional sample extracts from the same set of 20 samples. If the result from either of the additional spiked extracts is outside the acceptable range, use the method of additions for analysis of all 20 samples as described in EPA-600/4-79-020, March 1979.

f. Re-extraction of Sample Residue--For each 20 samples, extract and analyze one sample residue consisting of the filter paper and the undissolved soil portion using the usual digestion procedure. The parent sample will be X_{12}.

Calculate the percent of lead extracted by the first extraction based on the total of two extractions as follows:

$$\% \text{ extracted} = \frac{A}{A + B} \times 100$$

where: A = μg Pb/gm from first extraction

B = μg Pb/gm from second extraction (based on original sample weight).

Report results on the Soil Sample Quality Assurance Form.

g. "Total Recoverable" Analysis--For each 10 samples, a "total recoverable" analysis must be performed. The parent samples will be X_8 for g_1 and X_{20} for g_2. The extraction method chosen for this step has been demonstrated to give equivalent results for a variety of soil types when compared to known "total" methods. However, it is still necessary to closely monitor the extraction efficiency throughout this study. Once sufficient data have been gathered, re-evaluation of the frequency of the "total recoverable" analysis may be considered. It is expected that the results from the "total recoverable" method detailed below will agree within 10 percent with those generated using the standard method detailed in the section entitled Extraction Procedure. If after the analysis of 200 samples this agreement can be verified then the frequency of the "total recoverable" analyses might be lowered.

Note: The more rigorous HF-$HClO_4$-HNO_3 method which is widely used for total metals analysis in soil was considered but not chosen. This method is overly cumbersome, time-consuming and requires special equipment, and it was felt that any additional information that may be gained using the HF method would not warrant the additional effort.

h. Digestion Procedure--Weigh and transfer to a 150 ml beaker a 5.0-gram portion of sample which has been ground to pass a 100 mesh sieve. Add 25 ml of 1:1 HNO_3 and cover with a watch glass. Heat the sample at 95°C and reflux to near dryness. Allow the sample to cool, add 10 ml of conc. HNO_3 and again reflux to near dryness. After the second reflux step has been completed and the sample has cooled, add 5 ml of 1:1 HNO_3 and 3 ml of 30 percent hydrogen peroxide (H_2O_2). Return the beaker to the hot plate for warming to start the peroxide reaction. Care must be taken with the start of effervescence that losses do not occur or the reaction is not too vigorous. Heat until effervescence subsides and cool the beaker. Continue the addition of 30 percent H_2O_2 in 1 ml aliquots with warming until the effervescence is minimal or the general sample appearance is unchanged. Do not add more than a total of 10 ml 30 percent H_2O_2.

Add 5 ml of 1:1 HNO_3, return the covered beaker to the hot plate and reflux for an additional 10 min. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 100 ml with 1:4 nitric acid. The sample is now ready for AA analysis.

Report results in μg Pb/gm on the Soil Sample Quality Assurance Form. If the "total recoverable" analysis results differ by more than 10 percent from the results obtained using the standard digestion procedure, EMSL-LV should be contacted immediately for assistance.

i. Laboratory Control Sample Analysis--Analyze one aliquot of NBS-SRM 1645, River Sediment, for each 20 field soil samples, as an independent certified control sample.

Prepare and analyze the 5-gram samples in the same manner as project samples but eliminate the grinding and sieving steps.

Report the results on the Soil Sample Quality Assurance Form and calculate the percent recovery of NBS certified concentration using the following equation:

$$\% \text{ recovery} = B/A \times 100$$

where: A = NBS SRM certified lead concentration

B = analyzed lead concentration

Maintain control charts on the percent recovery and establish through repetitive analyses the 99 and 95 percent confidence limits (control and warning).

If the percent recovery is outside the acceptable range of 90 to 110 percent, or outside the 99 percent confidence limits, whichever is less, repeat the analysis. If the percent recovery is outside the acceptable range with the repeat sample, a thorough check of the extraction and analysis procedures should be conducted before proceeding with additional field sample analyses. EMSL-LV should be contacted immediately for assistance.

j. Split Extract Analysis—Intralaboratory precision for the analysis only will be estimated from the results of two replicate aliquots (split extracts) from every twentieth sample:

For each 20 soil sample extracts, split the extract from sample X_8 into two portions. Analyze the first portion as extract X_8, the second portion as i following X_{18}.

Report the results of the split extract analysis on the Soil Sample Quality Assurance Form.

A precision of 3 percent is expected. If the precision is outside of the 5 percent range, EMSL-LV must be notified immediately.

k. Spiked Sample Analysis--In order to monitor potential lead losses during extraction, perform a spiked sample analysis on a duplicate portion of one sample for each 20 field samples. Once sufficient data have been gathered, the frequency of the spiked sample analysis may be reconsidered.

For each 20 samples, spike a duplicate portion of sample X_g with 700 µg Pb, as lead nitrate solution, per gram of sample, and process through the normal digestion and analysis procedures. EPA may request a change of the amount of lead added during the course of the study.

Calculate the percent recovery of the spike as follows:

$$\%\ \text{recovery} = \frac{A - B}{C} \times 100$$

where: A = µg Pb/gm determined in spiked sample

B = µg Pb/gm determined in unspiked sample

C = µg Pb/gm added.

Report results on the Soil Sample Quality Assurance Form.

Maintain control charts on the percent recovery and establish through repetitive analyses the 99 and 95 percent confidence limits (control and warning).

No limits can be set at this time due to unknown sample homogeneity. However, if the percent recovery is outside of the 99 percent control limits, repeat the spiked sample analysis on a second sample from the same set of 20 samples. If this sample recovery is also outside of the 99 percent control limits, contact EMSL-LV immediately for assistance.

PROCEDURE

1. Grinding and Sieving--The laboratory will receive soil aliquots consisting of approximately 60 g of soil which has been previously dried, screened, mixed and split. The samples will be contained in 27-ounce whirl-pak polyethylene containers. Prepare the soil samples for extraction using the following procedures:

   a. Hand-mix each soil sample in its receiving container by tumbling the container 3 to 5 times.

   b. Grind approximately 5 grams of sample with a mortar and pestle (Note 2) sieve through a 100-mesh sieve, and weigh into a tared beaker. Dry for 1 hour at 105°C, cool, and weigh to the nearest 0.1 mg to determine the exact sample weight. Store the remaining original sample in the original container in a locked room until written permission has been received from EPA stating that the samples can be discarded.

Note 1: After every use, mortar, pestle and sieve shall be cleaned as follows: Wipe off all soil with tissue paper. Rinse three times with DI water. Wipe dry with tissue paper. When not in use, mortar, pestle and sieve must be protected from dust and fumes by storage in a closed glassware cabinet.

Note 2: The sample size of 5 grams was chosen over 1 gram because the larger sample size will reduce errors due to sample inhomogeneity.

c. Cover the beaker containing the weighed sample with a watch glass and keep it in a secured area until the extraction is performed.

2. Digestion

a. Add 50 ml of 1:1 HNO_3 to each soil sample and re-cover the beaker with a watch glass.

b. Heat the sample at 95°C and gently boil for 2 hours. Stir the sample occasionally with a glass rod. If during this step the volume goes below 25 ml, add more 1:1 HNO_3.

c. Allow the sample to cool, add 10 ml of 1:100 (v/v) HNO_3 and filter through Whatman No. 42 filter paper (or equivalent) into a 100-ml volumetric flask. Rinse the watch glass, glass rod, beaker and filter paper with two 10-ml portions of 1:100 HNO_3 and collect rinsings in the volumetric flask. Bring to volume with 1:100 HNO_3. The sample is now ready for analysis.

3. Atomic Absorption Analysis for Lead

a. Install the lead hollow cathode lamp. Set the wavelength of the monochromator at 283.3 nm. Align lamp and burner head. Background correction is required for these analyses.

b. At the beginning of each analysis set, but at least once per shift, calibrate the instrument using the working standards. Immediately following calibration, aspirate a known control sample such as the EPA Trace Metal QC Samples—WP475, independent of the calibration standards. Enter all information regarding instrument calibration in the instrument log book.

c. Aspirate sample extracts into flame. Take the mean of three integrations per sample (integration time = 5 to 7 seconds) and record this value (equilibrium absorbance) and the sample number on the strip chart tracing (or equivalent). Dilute all samples that exceed the highest working standard 1:10, with 1:4 HNO_3 so that their absorbances are within the linear working range. If the 1:10 dilution is not sufficient then dilute the original extract 1:100 with 1:4 HNO_3.

d. After each 10 samples aspirate one calibration standard of 10 μg/ml. If the standard result deviates by more than 5 percent from the value predicted by the calibration curve, recalibrate and repeat the previous 10 analyses.

e. After every 20 samples aspirate the known control sample as above.

4. Data Reduction and Reporting--Express soil sample analytical results as micrograms of lead per gram of soil (dry weight) and calculate using the following equation:

$$\mu g\ Pb/gm\ (dry\ weight) = ABC/D$$

where: A = μg/ml of Pb in sample extract

B = Final volume of sample extract (100 ml)

C = Dilution factor (10 or 100), if necessary

D = Sample weight in grams (dry weight).

The minimum reportable level is 10 μg Pb/gm based on the lowest working standard of 0.5 μg Pb/ml. Samples whose extracts are below 0.5 μg Pb/ml but are above the limit of detection (approximately 0.2 μg Pb/ml) should be reported but the values should be bracketed ( ), indicating that the results were detected but are below the lowest working standard. Samples at or below the detection limit are reported as ND (Not detected). Report results on the Sample Analysis Report Form (Figure 1) together with all required QC data (Figure 2).

REFERENCES

1. EPA Procedure for Soil Analysis, Dallas Lead Study, Revision No. 2, 6/30/82

DALLAS LEAD SMELTER STUDY
SOIL SAMPLE ANALYSIS REPORT

Report to: ______________________ Date reported: ______________

Distribution: ______________________

____

____

Laboratory Name: ______________________________

Date Sample Received	Field Sample ID	Lab Sample ID	Date Extraction Performed, Analyst Initials	Date Analysis completed, Analyst Initials	Result -------- µg Pb/gm Soil

____
Laboratory Supervisor

Figure 1.

SOIL SAMPLE QUALITY ASSURANCE

Report to: ______________________ Date reported: __________

Distribution: ______________________ QC Report No. __________

______________________ Sample Nos.; (Sequence as Analyzed)

____

____

____

Laboratory name: ______________________

Laboratory supervisor: ______________________ Analyst(s) initials: ______

---

1. Reagent blank: μg/ml ______________________

____

2. Field blank: μg/ml ______________________

Sample No. __________

3. Calibration control sample: μg/ml

Found ______________________

True ______________________

Source: __________

% recovery ______________________

4. Spike extract analysis: μg/ml

Sample No. __________

Sample extract result ______________________

Spike extract result ______________________

Spike added ______________________

% recovery ______________________

5. Residue re-extraction analysis: μg/gm

Original extraction ______________________

Residue extraction ______________________

Sample No. __________

% originally extracted ______________________

Figure 2.

---

6. Total recoverable analysis: µg/gm

Sample No. ____________________

Original result ____________________

"Total" method result ____________________

7. Laboratory control sample analysis: µg/gm

Source: NBS SRM 1645, River Sediment

Found ____________________

True ____________________

% recovery ____________________

8. Split extract analysis: µg/gm

Sample No. ____________

Original extract result ____________________

Split extract result ____________________

9. Spiked sample analysis: µg/gm

Sample No. ____________

Spiked sample result ____________________

Unspiked sample result ____________________

Spike added ____________________

% recovery ____________________

Figure 2 (continued)

PART 2. MAINTENANCE PROCEDURES AND SCHEDULE FOR MAJOR INSTRUMENTATION

GCA/Technology Division follows an orderly program of positive actions to prevent the failure of equipment or instruments during use. In the analytical laboratories, preventive maintenance includes attention to glassware, water supply, reagents and analytical balances as well as more complex instrumentation. Table 1 summarizes the preventive maintenance procedures for the instruments to be used in this program; also listed in the table are the spare parts normally kept in inventory to minimize instrument downtime.

All information regarding maintenance or repair procedures is entered in the instrument log book.

TABLE 1. MAINTENANCE PROCEDURES AND SCHEDULE FOR MAJOR INSTRUMENTATION

Instrument	Maintenance procedure/schedule	Spare parts
Analytical Balance[a,b] Mettler H80, HL32	1. Service, cleaning by Mettler Technician--annually 2. Clean weighing pan immediately if any material is spilled. Brush pan to remove dust before each use. 3. Check balance level before each use. 4. Check calibration using Class S weights before each use and when balance is moved.	1. Light bulbs 2. Fuses (HL32)
Atomic Absorption Spectrophotometer, Perkin-Elmer 460,[c] Varian AA-6[d]	1. Clean optical surfaces weekly. 2. Clean burner head and mixing chamber, as needed. 3. Clean nebulizer and sample capillary as needed.	1. Fuses 2. Spare nebulizer assembly.

[a] Mettler Manual for the H80 Analytical Balance, Mettler Instrument Corp., Hightstown, NJ, June 1978.

[b] Mettler Manual for the Model HL32 Analytical Balance, Mettler Instrument Corp., Hightstown, NJ, March 1978.

[c] Model 460 Atomic Absorption Spectrophotometer Instruction Manual, Perkin-Elmer, Norwalk, CT, December 1975.

[d] Model AA-6 Atomic Absorption Spectrophotometer, Varian Techtron, Ltd., Melbourne, Australia, May 1973.

WORK PLAN SUMMARY (WP-1)

FOR PROJECTS SUPPORTED BY
INDUSTRIAL ENVIRONMENTAL RESEARCH LABORATORY
OFFICE OF RESEARCH & DEVELOPMENT/ENVIRONMENTAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NORTH CAROLINA 27711

Project title: ANALYSIS OF DALLAS SOIL SAMPLES

Contractor name, division: GCA CORPORATION, GCA/TECHNOLOGY DIVISION

Contractor project manager: Dr. Kenneth McGregor — Phone: (617) 275-5444

Address: 213 Burlington Road (Street) — Bedford (City) — MA (State) — 01730 (Zip code)

EPA project officer: Alice C. Gagnon

1 Contract No.: 6 8 0 2 3 1 6 8	9 Task/TD No.: 80 - (3)	12 Date work plan submitted (M M D D Y Y): 0 7 2 0 8 2	18: B
19: 01	21 Project start date (M M D D Y Y): 0 7 1 4 8 2	27 Project completion date (M M D D Y Y): 1 0 3 0 8 2	Check one: 33 ☒ Original 34 ☐ Revision

Project location: 35 State: M A — 37 Zip code: 0 1 7 3 0

Bedford (City) — Middlesex (County) — USA (Country)

Project type:

Reason for revision:

Costs: Estimated contract cost — Contract fee

Project objective (50 typed characters · elite · 12 pitch).

19 03 21 The primary objective of this task is to provide
04 support to the EPA in a study of elemental lead in
05 soil, blood, dust, and vegetation samples collected
06 in the Dallas, Texas area. GCA will operate a
07 Sample Bank in Dallas for receiving and disburse-
08 ment of samples to appropriate analytical labora-
09 tories. The GCA laboratory will perform analysis
10 of elemental lead in soil samples using AAS and
11 will conduct a study on soil sample homogeneity.
12
13
14

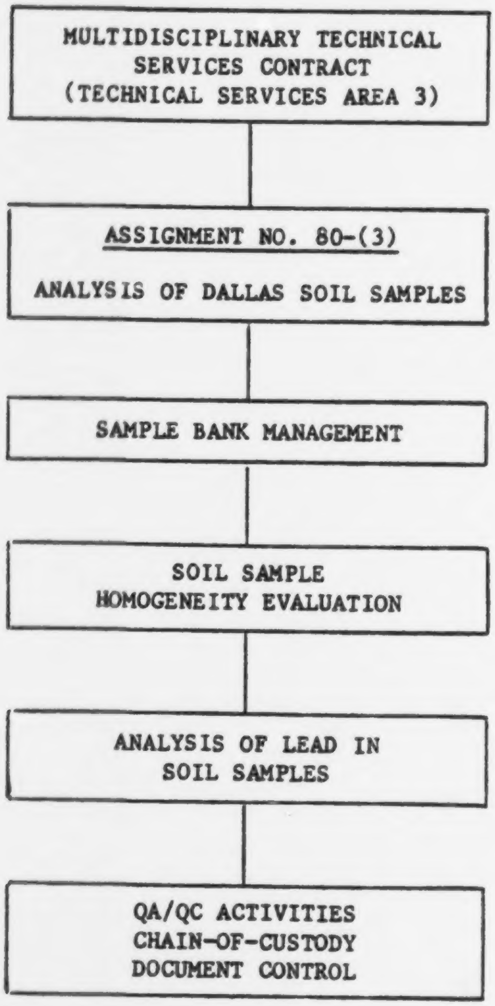
77 ☐ a 78 ☐ b 79 ☐ c 80 ☐ d

DO NOT WRITE IN THIS SPACE

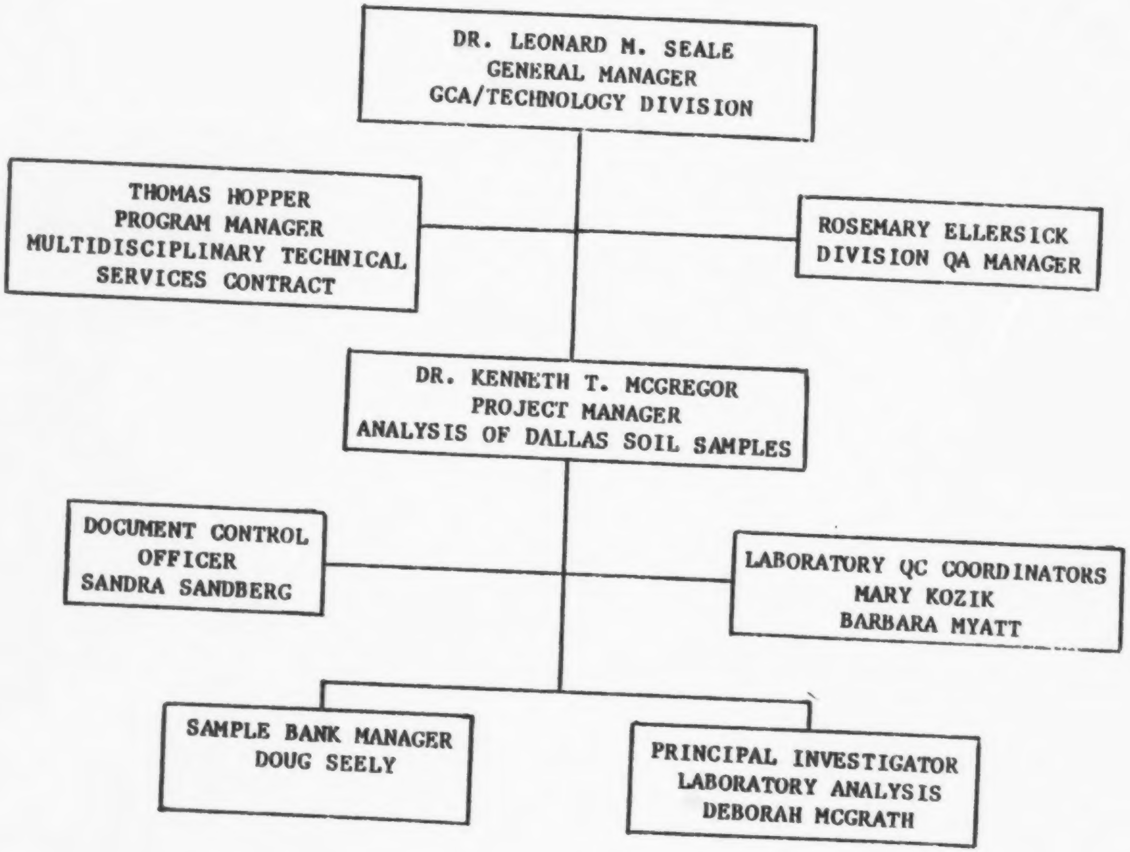
EPA (RTP) 398

BEST COPY AVAILABLE
FOR CLARIFICATION CONTACT
KENNETH W. BROWN (702) 798-2214

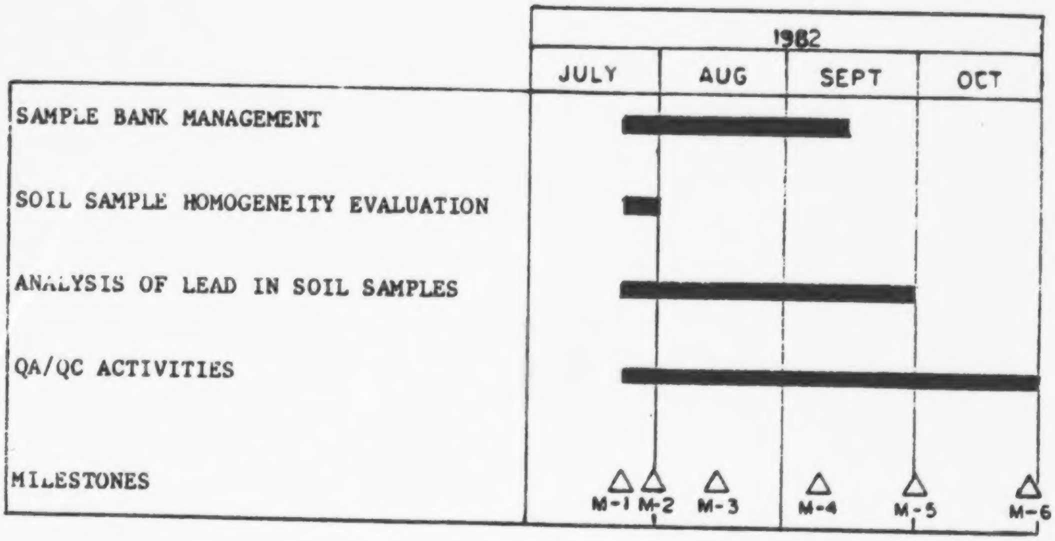
2.0 WORK BREAKDOWN STRUCTURE


MULTIDISCIPLINARY TECHNICAL SERVICES CONTRACT (TECHNICAL SERVICES AREA 3)
ASSIGNMENT NO. 80-(3)
ANALYSIS OF DALLAS SOIL SAMPLES
SAMPLE BANK MANAGEMENT
SOIL SAMPLE HOMOGENEITY EVALUATION
ANALYSIS OF LEAD IN SOIL SAMPLES
QA/QC ACTIVITIES
CHAIN-OF-CUSTODY
DOCUMENT CONTROL

3.0 PROGRAM ORGANIZATION AND RESPONSIBILITY


DR. LEONARD M. SEALE
GENERAL MANAGER
GCA/TECHNOLOGY DIVISION
THOMAS HOPPER
PROGRAM MANAGER
MULTIDISCIPLINARY TECHNICAL
SERVICES CONTRACT
ROSEMARY ELLERSICK
DIVISION QA MANAGER
DR. KENNETH T. MCGREGOR
PROJECT MANAGER
ANALYSIS OF DALLAS SOIL SAMPLES
DOCUMENT CONTROL
OFFICER
SANDRA SANDBERG
LABORATORY QC COORDINATORS
MARY KOZIK
BARBARA MYATT
SAMPLE BANK MANAGER
DOUG SEELY
PRINCIPAL INVESTIGATOR
LABORATORY ANALYSIS
DEBORAH MCGRATH

4.0 PROJECT SCHEDULE



	Milestones	Date Planned
M-1	Work Plan and QA Project Plan Submitted	07/23/82
M-2	Soil Homogeneity Evaluation Completed	07/30/82
M-3	QA Audit of Laboratory Analyses	08/15/82
M-4	Shipment of Samples Completed (tentative)	09/10/82
M-5	Analysis of Soil Samples Completed (tentative)	10/01/82
M-6	Final Report Submitted	10/30/82

COST/SCHEDULE BASELINE (WP-2)

Page 1 of 1

Contractor GCA/TECHNOLOGY DIVISION
GCA CORPORATION

Contractor project manager name, title K. McGregor
Head, Laboratory Analysis Department

Revision No

Date

EPA project officer Sandra D. Lee

K McGregor
Signature

68-02-3168 80(3)
Contract No Task No

27 July 1982
Date

Approval by EPA project officer[a]

(1) Work breakdown structure		(2) Cumulative cost to date ($1000's)[b]	(3) Monthly budgeted cost of work scheduled (BCWS)[b, c] ($1000's)																(4) Total budget ($1000's)[b]
No.	Noun description		Mo.	Yr.	Mo.	Yr.	Mo.	Yr.	Mo.	Yr.	Mo.	Yr.	Mo.	Yr.	Mo.	Yr.	Mo.	Yr.	
			7	82	8	82	9	82	10	82									
	WA No. 80	0	18.0		36.0		31.5		29.0										114.5
General and administrative		0	0		0		0		0										0
Management reserve																			
Total		0	18.0		36.0		31.5		29.0										114.5
Cumulative total cost *(excluding fee)*		0	18.0		54.0		85.5		114.5										

[a] Once the cost/schedule baseline has been approved, the figures may not be changed without the approval of the EPA project officer.

[b] All costs shown are exclusive of fee.

[c] Montly budgeted costs for entire contract–attach additional sheets if necessary.

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6.0 PROJECT DESCRIPTION

6.1 Technical Objectives

The major objective of this project is to provide support to the EPA in a study of elemental lead in soil, blood, dust and vegetation samples collected in the vicinity of Dallas, Texas. These environmental and biological samples will be analyzed for lead to determine whether elevated blood lead levels exist and whether they can be attributed to present or past emissions from lead smelters operating in the area.

6.2 General Approach

GCA's responsibilities in this study include establishing and operating a Sample Bank in Dallas and preparing and analyzing soil samples for lead using flame atomic absorption spectrometry (AAS). The Sample Bank will be set up to receive samples, maintain custody and shipping records, and distribute samples to the appropriate analytical laboratories. Soil samples will be analyzed at GCA using protocols provided by EPA. An initial shipment of replicate soil samples will be analyzed to determine whether field preparation procedures are capable of providing homogeneous samples.

The major work elements required for the performance of this task are described below.

6.2.1 Sample Bank Management--GCA will operate the Sample Bank in Dallas which will be the central receiving, shipping and storage facility for all samples associated with the Dallas Lead Investigation. Current estimates include 5000 soil samples, 1000 dust samples, 1200 blood samples, and 3000 vegetation samples. All equipment for processing samples is being supplied by EPA; GCA's role will be to manage the overall operation of the sample bank and to provide technical staff for grinding, partitioning and packaging samples for shipment to analytical laboratories for subsequent lead analysis. GCA staff will also supervise the implementation of chain-of-custody procedures for all samples generated for the program; this will include field blanks and other QA samples.

The soil sampling protocol has been developed by EPA and is included in this Work Plan as Appendix A - "Dallas Lead Investigation - Soil Sampling Protocol." Responsibility for collecting samples will be assumed by EPA using manpower to be supplied by the City of Dallas. For sample numbering, identification and logging purposes, GCA will interact directly with the EPA sample crew supervisor.

Procedures for operating the Sample Bank have developed by EPA and are included in Appendix A under the heading Attachment 2 - "Sample Bank Procedures." These will be implemented by GCA immediately upon assuming responsibility for the Sample Bank.

The goal for sample bank operations is to process about 120 samples per day over a 7-week period. The first priority is to process the existing backlog of several hundred samples. Sample throughput is highly dependent on equipment availability, with critical items being a rolling mill for mixing samples and an oven for drying samples prior to shipment. At present, there is only one of each available to the program. To optimize scheduling and equipment availability, GCA will use staggered shifts where appropriate. The rate of sample collection and the delivery schedule to the Sample Bank may need adjustment in order to optimize throughput. An acceptable schedule should be developed within the first week of the project.

6.2.2 Soil Sample Homogeneity/Extraction Efficiency Evaluation--Sample handling procedures for this task require that soil samples be dried, sieved, mixed and repackaged by Sample Bank personnel prior to shipment to the analytical laboratory. To evaluate the ability of this procedure to provide homogeneous samples, a preliminary study will be conducted on replicate samples. An additional part of this preliminary study is an evaluation of the extraction efficiency of the prescribed sample preparation technique.

Eleven (11) samples will be split into three (3) aliquots each in the field and submitted to the GCA laboratory for analysis. Sample handling, preparation, and analytical procedures for these 33 samples will be those provided by EPA for use throughout the program. In addition, one extraction residue of one of the three split portions will be subjected to the "Re-extraction of Sample Residue" procedure provided by EPA and a second 5-gram aliquot of the same split portion will be subjected to a "Total Recoverable Analysis." QA/QC procedures specified for the soil analysis program will also be used in this evaluation.

Primary analytical and QA/QC protocols are briefly described in Section 6.2.3 of this Work Plan. The complete protocols for routine analysis and extraction efficiency testing (provided by EPA) are included as Appendix B to this Work Plan. The QA Project Plan gives additional details of QA/QC activities to be conducted throughout the task and will be submitted under separate cover.

Results of lead analyses performed as part of the homogeneity study will be submitted promptly to EMSL-LV for evaluation. Proposed action limits established by EPA are as follows:

- If the split sample analyses precision exceeds 15 percent then the Sample Bank protocol may be tightened to include additional blending and/or grinding.
- If the "Total Recoverable" results are greater than 10 percent from the Protocol Method results (including the re-extraction data) then EMSL-LV may consider some protocol modifications.

6.2.3 Lead Analysis of Soil Samples--GCA will analyze approximately 5000 soil samples (up to 700 per week) for elemental lead using analytical and QA protocols provided by EPA. These procedures are summarized below and complete protocols are provided in Appendix B to this Work Plan and in the QA Project Plan submitted under separate cover.

Soil samples will be received by the laboratory as 60-gram aliquots that have been dried, screened, mixed and split at the Sample Bank in Dallas. At GCA, the sample will again bemixed in its receiving container and a 5-gram aliquot removed for analysis. Preliminary treatment of this sample will include grinding, sieving, and weighing before and after drying at 105°C. The sample will then be extracted in a covered beaker with 1:1 HNO_3 by gently boiling at 95°C for 2 hours. After cooling, 10 ml 1:100 HNO_3 will be added to the extract and the whole solution will be filtered through Whatman No. 42 (or equivalent) filter paper into a 100 ml volumetric flask. Prescribed rinsings of the extraction apparatus and filter paper will be made with 1:100 HNO_3, and the solution will be made to volume with 1:100 HNO_3.

Analysis of elemental lead in soil extracts will be performed by flame AAS using a hollow cathode lamp source and the 283.3 nm wavelength. The use of background correction is required by the EPA protocols. The instrument will be calibrated using at least six calibration standards ranging in concentration from 0.5 to 15 μg Pb/ml and one calibration blank consisting of 1:4 HNO_3. Procedures for calibration will be those recommended by the instrument manufacturer. A calibration curve of absorbance versus concentration will be prepared for quantification of lead in sample extracts. Linear regression analysis will be used to determine the linear working range of the calibration curve. A known QC sample such as the EPA Trace Metals in Water QA Sample will be used to verify proper calibration before any samples are analyzed.

QC procedures to be applied to the AAS analyses include the analysis of a check calibration standard (10 μg Pb/ml) after every 10 samples. If this standard is not within 5 percent of the expected value, the instrument must be recalibrated and the previous 10 samples must be reanalyzed. After every 20 samples, a known QC sample (EPA Trace Metals in Water) must be analyzed and the results reported as part of project quality assurance.

Soil sample analytical results will be reported as micrograms of lead per gram of soil (dry weight basis). Calculations will be performed according to the equations provided in the EPA protocols. The minimum reportable level has been determind to be 4 μg Pb/gm.

APPENDIX A

SOIL SAMPLING PROTOCOL
SITE I.D. SOIL GRID SAMPLING SITES
SAMPLE BANK PROCEDURES

Section No. 1
Revision No. 5
Date 7/7/82
Page 1 of 13

DALLAS LEAD INVESTIGATION

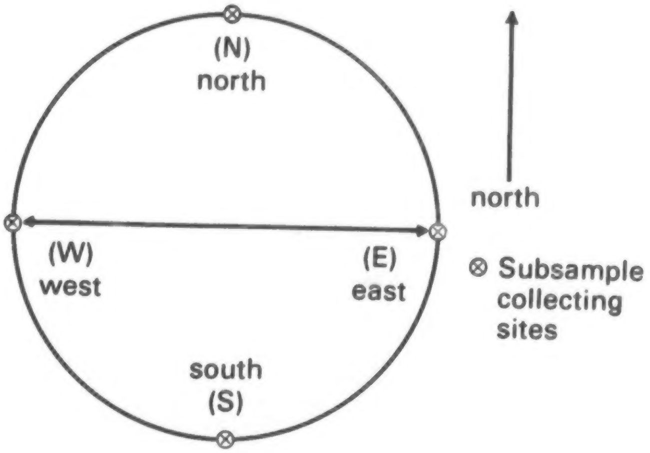
SOIL SAMPLING PROTOCOL

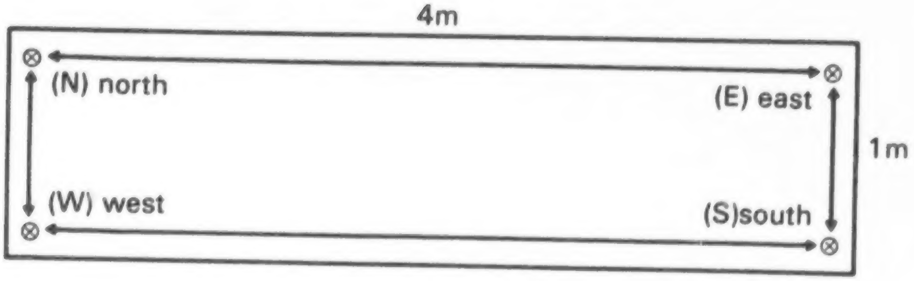
A. Location

1. U.S. Environmental Protection Agency (EPA) personnel from the Environmental Monitoring Systems Laboratory at Las Vegas (EMSL-LV) and/or the U.S. Public Health Service (PHS) personnel from the Center for Disease Control (CDC) at Atlanta will designate and identify all sampling locations.

2. Samples will be obtained by compositing subsamples of approximately 40 g each collected from four (4) equally spaced locations on the arc of a ten (10) meter diameter circle. One subsample will be collected at the northernmost point on the arc, one at the southernmost point, one at the easternmost point and one at the westernmost point.

Some of the designated soil sampling locations will not be large enough in area to collect soils around the required ten (10) meter diameter circle. The reduction of the diameter of the circle by one (1) meter increments to a minimum circle diameter of three (3) meters will be acceptable. If a sampling circle, as illustrated below, is not possible, measure and collect four (4) subsamples from a rectangular configuration as shown in the second diagram.





Soil collected from the four subsampling sites, located in each corner of the rectangle will be composited identical to those collected from the sampling circle.

3. Two methods of locating soil sampling locations will be used:

(1) A square sampling grid measuring 750' by 750' will be used to select sampling locations within and covering a 1 mile radius from both the RSR Corporation (RSR) and Dixie Metal Company (DMC) lead smelter sites. The grid will also be used to identify sampling locations at a reference (REF) site. The dimensions of the grid at the REF site will be identical to that used at the RSR and DMC sites. The area sampled at the REF site will only include a one-half mile radius from the intersection of Frio Drive and Kiest Boulevard.

The axes of the grid at the RSR, DMC and REF sites will run north-south, and east-west. The exact sampling locations will be identified on aerial photographs for each of the three areas. Samples will be collected near each grid intersection via the circular or rectangular configurations previously described.

(2) Specific locations at schools, parks, playgrounds, and/or in yards will be selected for sampling. These sampling locations will be selected by CDC personnel on a house-by-house and location-by-location basis to complement blood lead data. The sampling locations will primarily be contingent upon the activity patterns of preschool-aged children. Other criteria used to select soil sampling locations will be identified by CDC personnel. Soil samples will be collected at each of these locations via the circular or rectangular configurations previously described.

B. Frequency

1. A total of 177 soil sampling locations will be selected from the 750' by 750' grid intersections at each of the two (RSR and DMC sites. Approximately 89 sampling locations will be selected at the REF site.

Approximately 1,200 additional sampling locations will be selected at specific homes, parks, schools and playgrounds. At five (5) percent of the sampling locations duplicate samples will be collected. At an additional five (5) percent of the sampling locations the samples collected will be split into three homogeneous portions. At the duplicate sampling locations, the duplicate subsamples will be collected within a 0.5 meter radius of the original subsample collection location. The duplicate subsamples will be composited separately and prepared identical to those collected at the original subsite locations.

C. Pollutant Class

The soil samples will be collected for the analysis of lead.

D. Field Blanks

Field blanks are used to monitor sample handling procedures, container cleanup, and to document cross contamination. Field blanks are to be prepared in the field and handled in the same manner as collected samples. Field blanks will be placed and remain in the field sample storage container during the daily field sampling effort.

The field blank will be analyzed by the laboratory analyzing the samples, therefore, a field blank is required for each type of sample collected and for each media (soil and dust) collected. Unless otherwise stated, the field blank will be DDI (distilled deionized) water. The sampling teams as selected by sample bank personnel shall transport DDI water to the sampling locations and prepare one field blank for every 20 soil samples collected. The field blank will be DDI water that has come in contact with the soil corer.

E. Sampling Procedures

1. Container

The soil samples are to be contained in sealable polyethylene containers that have the capacity to hold 200 g. The samples are to be kept in a covered container at ambient meteorological conditions until delivered to the sample bank.

2. Container Preparation

The containers will be sterile 27 ounce whirl-pak polyethylene containers. As such, precleaning will not be required. These sample containers should be stored and kept in a closed container to minimize atmospheric contamination.

3. Sample Collection and Handling Procedures

A nickel chrome-plated soil corer with an inside diameter of 0.75 inches, and capable of a vertical penetration into mineral soil 3 inches deep, shall be used to extract the four 3-inch deep core subsamples. The composite samples will consist of all four subsamples placed in the same sample container.

The sample shall consist only of mineral soil. If sod layers are encountered, such as grass, remove the surface vegetative sod material by dissection using a scalpel. Discard the scalpel blade and replace with a clean blade after sampling at all four subsites. Pre-cleaned polyethylene gloves must be worn by the sampler to avoid sample contamination during collection and/or dissection.

Place any vegetative material in a clean polyethylene container and attach a properly filled out vegetative collection tag to the container. Vegetative subsamples will be composited identical to soil subsamples.

The senior member of the field sampling team will be responsible for adhering to the following sample collecting guidelines.

1. At designated grid sampling locations the sampling site configuration (circle or rectangle) shall be located at the exact grid intersection point. If this location is unsatisfactory due to streets, structures, and/or other obstructions, the sampling site can be moved within a 30-meter radius of the grid intersection. If a sample cannot be obtained within the 30-meter radius the on-scene coordinator should be notified.

2. Avoid if possible collecting samples that are less than 20 feet from painted surfaces.

3. Locate collection sites as far as possible from vehicle activity such as streets, driveways, parking and automobile repair areas.

4. Avoid if possible collecting samples under or immediately adjacent to trees, shrubs, and/or structures.

5. Complete site description forms (See Part F).

6. Thoroughly wash and rinse the soil corer in hot tap water and then rinse the corer with DDI water after sampling at each location. A suitable brush or Kimwipes may be necessary for adequate cleanings. If hot tap water is unavailable, use cold tap water with a suitable lead-free laboratory detergent. Label the sample container with an appropriate sample collection tag (as shown) supplied by the sample bank.

<table>
<tr><td>Project Code
Dallas Lead</td><td>Soil</td><td>(Source of Sample)
Grid Park School
Home Playground Other</td><td colspan="3">(Site Code)</td><td rowspan="4">Tag No.</td></tr>
<tr><td colspan="3">Address of Site (or Location)</td><td>Date</td><td colspan="2">Time
(Military)</td></tr>
<tr><td colspan="6">Sampler (signature)</td></tr>
<tr><td colspan="4">Remarks:

Sample I. D. No. ____________________</td><td>Lead
XXXX</td><td>Analysis</td></tr>
</table>

Sample Collection Tag

The sample collection tags have been designed for soil, vegetation and dust samples. They are a different color for each of the three media and are serially numbered starting with a different block of numbers for each media and for each (RSR, DMC and REF) site. The tags are preprinted to ensure that the required information is provided on each tag. Each collected sample, including duplicates and field blanks, shall have a completely filled-in sample tag securely attached to it. Duplicates and field blanks shall be identified in the "remarks" section of the tag and by a specific sampling site identification code number. See Site I. D. Soil Grid Sampling Sites, Attachment 1. Samples collected for splits shall be identified in the remarks section of the tag.

The person who physically collects the sample is the SAMPLER and signs the sample tag. The SAMPLER initiates the custody record for transfer of samples to the sample bank.

After the samples have been transferred to the Sample Bank and documented there, they will be renumbered with a sample bank number and identified with a new sample analysis tag as shown below. This renumbering step enables the submittal of duplicate samples and QC samples as blinds to the analytical laboratory.

(Project Code) Dallas Lead	(Sample Bank No.)
(Sample Description)	
Analysis	
Metals (Lead)	
(Tag No.)	

Analysis Tag

The original sample collection tag number is recorded in the sample bank's master log and the tag itself is stored in the document file at the Sample Bank (see Attachment 2, Sample Bank Operation). For vegetation samples only leave the sample collection tag on the sample container. Traceability of samples from collection location to laboratory analysis is required as such, cross indexing of all sample collection tag numbers, and corresponding sample bank and analytical tag numbers must be made in the master logbook.

The sample bank will also be responsible for drying and mixing each soil sample. This drying and mixing procedure to achieve homogeneity will be accomplished by placing the total soil sample in a clean 250 ml glass beaker. Place the beaker in a drying oven at a temperature of 100°C for an 8-hour period or until the soil sample is dry. After drying, place the soil in an 8" diameter stainless steel 16 mesh sieve. Sieve the soil through the sieve with the aid of a glass pestle. Discard the unsieved portion. Place the sieved portion in a lead-free porcelain grinding jar and mix by placing the jar on a multi-tiered jar mill and rotate at a speed of 250 rpm for a 30-minute period. After the 30 minute mixing period place 1/3 or approximately 50 g of the dry soil in a clean polyethylene sample container and attach a properly filled out sample analysis tag. The remaining 2/3 of the soil sample shall be placed in a clean polyethylene sample container and properly labelled so that it can be identified as being part of the same sample as the analyzed (1/3) portion. The 2/3 portion shall be stored at the sample bank.

The vegetative samples shall be placed in a freezer at 0°C and stored at the sample bank. Analysis of the vegetative tissue may be required at a later date. Those soil samples identified as splits on the sample collection tag will be dried and mixed and then split into three equal but separate samples for analysis. One of the split samples will be shipped to the referee analytical laboratory. The remaining two split samples will be shipped to the same analytical laboratory. The identification of these split samples must be documented in the master log. Samples identified as duplicates on the sample collection tag will also be shipped to the same analytical laboratory.

The equipment used for drying and mixing the soil must be decontaminated after each sample. Wash and rinse each component of the drying and mixing equipment in hot tap water and then rinse with DDI water. Use a lead-free laboratory detergent and laboratory brushes or Kimwipes in the decontamination procedure if required.

F. Site Description Form

An example of the site description form that will be used in this study is shown as Figure 1. The senior member of the field sampling team will be responsible for completing one site description form for each location sampled. Documentation at each site should include appropriate photographs showing the area sampled. In addition, a schematic drawing showing sampling locations, including dimensions and other pertinent information such as structures, ground surface characteristics, streets and driveways will be prepared.

The site description forms will be deposited and stored at the sample bank.

G. Sample Transfer Procedures/Chain-of-Custody

There will be a number of transfers of custody in this program. Samples are to be delivered to the sample bank by the sampler. The samples will be shipped to analytical and referee laboratories by the sample bank. In addition Quality Control (QC) samples, provided by other laboratories will be shipped to the sample bank for inclusion with collected environmental (soil, vegetation, dust and blood) samples. All entities that generate samples for this study are required to initiate custody records.

Customized chain-of-custody record sheets, following the NEIC format will be provided by the sample bank. Examples of these record sheets are shown as Figures 2 through 4. Figure 2 is used for samples collected in the field, Figure 3 is for shipping QC samples to the sample bank and Figure 4 is used for shipping samples to analytical laboratories.

The custody record sheets will be used for a packaged lot of samples; more than one sample will usually be recorded on one form. More than one custody record sheet may be used for one package, if necessary. Their purpose is to document the transfer of a group of samples traveling together; when the group of samples changes, a new custody record is initiated. The original of the custody record always travels with the samples; the initiator of the record keeps a copy. When custody of the same group of samples changes hands several times, some people will not have a copy of the custody record. This is acceptable as long as the original custody record shows that each person who had received custody has properly relinquished it.

Using the Custody Record Sheet

- The originator fills in all requested information from the sample tags.
- The person receiving custody checks the sample tag information against the custody record. He also checks sample condition and notes anything unusual under "Remarks" on the custody form.
- The orginator signs in the top left "Relinquished by" box and keeps the copy.
- The person receiving custody signs in the adjacent "Received by" box and keeps the original.
- The Date/Time will be the same for both signatures since custody must be transferred to another person.
- When custody is transferred to the Sample Bank or an analytical laboratory, blank signature spaces may be left and the customized last "Received by" signature box used.
- In all cases, it must be readily seen that the same person receiving custody has relinquished it to the next custodian.
- If samples are left unattended or a person refuses to sign, this must be documented and explained on the custody record.

The sample bank will retain copies and/or original shipping papers, bill of lading and other records dealing with the shipment of samples.

If discrepancies between sample tag numbers and custody record listings are found, the person receiving custody should document this and properly store the samples. The samples should not be analyzed until the problem is resolved by contacting the on-scene coordinator or other designated responsible authority.

The responsible person receiving custody should attempt to resolve the problem by checking all available information (other markings on sample container, type of sample, etc.). He should then document the situation on the custody record in his project logbook and notify the on-scene coordinator as soon as possible.

Changes may be written in the "Remarks" section of the Custody record and should be initialed and dated. The copy of this record should accompany the written notification to the on-scene coordinator.

Custody Seals

Custody seals will be required when shipping samples by commercial carriers. The seals are narrow strips of adhesive paper used to demonstrate that no tampering has occurred. They are intended for use on a sample transport container which is not secured by a padlock. They are not intended for use on individual sample containers.

Custody of Shipped Samples

When a group of samples with its custody form is to be shipped, the shipper (e.g. sample bank personnel) accompanies the package to the carrier (e.g., Federal Express) so that, if requested, the number and identification of the samples in the container can be verified. The commercial carrier is not required to verify this.

The package is then closed with a strong, keyed padlock or strapping tape and custody seals so that the carrier is transporting a secure container.

NEIC's recommended procedure for custody seal use on shipping boxes is as follows:

- Place paper side of seal against box and wrap strapping tape across the middle of the seal and at least twice around the box.
- Cut the tape so that the ends overlap at the seal.
- Fold the seal to itself over this overlap and sign the custody seal.
- Repeat this procedure as many times as necessary to seal the box for shipment.

The person receiving custody of shipped samples must document the condition of the locked, or strapped and sealed shipping box on arrival. It must be checked that neither the tape or the custody seals has been cut or otherwise tampered with. If the paper seal has been damaged in shipping but it is clear that the shipping box has not been opened, further handling of the samples may proceed. If tampering is suspected, the designated Sample Custodian shall notify the on-scene coordinator and the sample bank supervisor.

PROJECT CODE: Dallas Lead.

DATE ____________

SAMPLER: ____________________

SITE ADDRESS: (STREET, NUMBER, COORDINATES) ____________________

TYPE OF SITE: (RESIDENTIAL, INDUSTRIAL, ETC.) ____________________

PROPERTY OWNER: ____________________

LOCATION OF SAMPLING: (BACKYARD, PARK, ETC.) ____________________

*SKETCH MAP OF THE SAMPLING AREA AND ATTACH IT TO THIS FORM.

ATTACH POLAROID PHOTOGRAPH OF SAMPLING SITE TO THIS FORM.

ADJOINING PROPERTY: (RESIDENTIAL, INDUSTRIAL, ETC.) ____________________

GROUND SURFACE: (BARE, LAWN, ETC.) ____________________

STRUCTURAL ODDITIES: (SWIMMING POOLS, SHEDS, ETC.) ____________________

TYPE OF SAMPLING PERFORMED: (SOIL, DUST, ETC.) ____________________

SAMPLE COLLECTION TAG NUMBERS: ____________________

CONDITION OF SAMPLING SITE: (DEBRIS, RESIDUES, STANDING WATER, ETC.) ____________

REMARKS: (INCLUDING ALL SAMPLE NUMBERS) ____________________

*Sketch of the sampling area should be prepared with fixed landmarks suitable as reference locations. Distances and locations must be measured by tape or hand-held range finding equipment, or a combination of both.

Figure 1. Sampling Site Description Form.

Figure 2.

Environmental Protection Agency
Office of Enforcement

CHAIN OF CUSTODY RECORD

Proj. No.

Project Name DALLAS LEAD

SAMPLERS: (Signature)

						No. of Containers	Analysis		Sample Type					
TAG NO.	**DATE**	**TIME**	**COMP.**	**GRAB**	**SITE LOCATION**		**LEAD**		**SOIL**	**DUST**	**BLOOD**	**VEGETATION**	**OTHER**	**REMARKS**

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Sample Bank by: (Signature)	Date/Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Originator Files

Figure 3.

Environmental Protection Agency
Office of Research and Development

CHAIN OF CUSTODY RECORD
QC SAMPLES

Proj. Code	Project Name DALLAS LEAD				Analysis	
TO: SAMPLE BANK	SAMPLE DESCRIPTION					REMARKS
Sample No.	Water	Soil	Sediment	Other	LEAD	

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Sample Bank by: (Signature)	Date/Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Originator Files

Figure 4.

Environmental Protection Agency
Office of Research and Development

CHAIN OF CUSTODY RECORD

Proj. Code	Project Name DALLAS LEAD				Analysis	
TO: ANALYTICAL LAB	SAMPLE DESCRIPTION					REMARKS
Sample No.	Water	Soil	Sediment	Other	LEAD	

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Laboratory: (Signature)	Date/Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

ATTACHMENT 1

SITE I.D. SOIL GRID SAMPLING SITES

I. Sampling Site Identification (Site Code)

Aerial photographs of each area (RSR, DMC and REF) overlayed by a 750' x 750' soil sampling grid will be provided to each sampling team. Every grid line is identified. The rows are identified alphabetically, and the columns numerically.

Each soil sample collected from the grid configuration will have a site identification code that will identify the grid site from which the sample was collected. It is required that all forms that originate with the sample collection (sample collection tags, site description forms and chain-of-custody forms) have a site identification code on them. Sample bank personnel will not accept any sample in which information, as required on the forms, is missing. The sampling team leader will be responsible for making sure all required sampling forms are properly filled out.

The grid sampling site identification code will be as follows:

a. The first three letters will identify the grid location, RSR, DMC or REF.

b. Place a hyphen - after the last letter of the grid location designation.

c. The fifth space will be a letter identifying the row (A through O).

d. The sixth and seventh spaces will be numbers identifying the columns (01 through 15).

e. A typical site identification code should appear as RSR-F08. At five percent (5%) of the grid sampling sites (listed below) duplicate samples will be collected. The duplicate (the second one collected) will be identified by adding an A immediately following the number identifying the column. For example:

   RSR-E03 identifies the first soil sample collected at the RSR location, at the grid intersection located in row E, column 03, while RSR-E03A identifies the duplicate sample collected at the same location, row and column.

Duplicate soil samples will be collected at the following row and column grid sampling sites: E03, E14, F12, G02, G15, H14, I05, I06, J11, K04. For every 20 soil samples collected from the grid sampling sites one field blank will be collected. The sample bank personnel will select and notify the team responsible for collecting the field blank on a daily basis as required. Field blanks will be identified with a specific soil sampling site. The field blanks will be collected immediately prior to collecting a soil sample at a designated site. For example, if the next soil sampling site is identified as RSR-F08, prepare the field blank first before collecting the soil sample. Identify the field blank by placing a B immediately following the number identifying the column; i.e., RSR-F08B.

The appropriate site identification code for each soil sample collected by grid designation including duplicate and field blanks must appear on the sample collection tag in the space identified as (site code) on the chain-of-custody form space identified as site location, and on the site description form space identified by site address.

Designations X, Y and Z immediately following the column identification number will indicate the soil sample as a split: i.e., RSR-D02X, RSR-D02Y and RSR-D02Z. The sample bank personnel however, will add this designation. Soil samples designated for splitting will be collected at the following row and column grid sampling sites: D02, E07, E09, F03, J07, K09, L06, M10, M13, N05.

The sampling teams will also be provided aerial photographs of each location that will enable the sampling team leader to plot and identify the exact sampling site.

ATTACHMENT 2

SAMPLE BANK PROCEDURES

I. Responsibility

The sample bank is the custodian for all records pertaining to the sampling, sample preparation as required, and shipment of environmental samples to analytical laboratories.

The sample bank is responsible for record filing and storing, for storing and preparation of soil and vegetation samples, and for dispensing containers, sampling equipment and all custody documents such as chain-of-custody forms and sample collection and analytical tags, as required.

The sample bank is responsible for updating and maintaining the projects' master log book, auditing the records as required, generating sample bank QC sample blanks, accepting QA/QC samples for inclusion into the analytical scheme, and for scheduling the collection of field sample blanks.

The sample bank is responsible for completing, as required, analysis data reporting forms and for assuring that all chain-of-custody requirements as stated in the Dallas Lead Studies Quality Assurance Plan, pertaining to all field sampling, shipping and sample bank operations, are adhered to.

II. Procedures

a. Issuing Supplies:

(1) On a daily basis, or as appropriate, the sample bank will issue as required sample containers, sample collection tags, (soil, dust, blood, and vegetation), chain-of-custody forms as shown on Figure 2, and site description forms to the sampling teams. As the sample collection tags and chain-of-custody forms are accountable documents, the sample bank will log the forms by numerical lot identifying the team and/or the individual responsible for the temporary custody of these documents.

(2) The sample bank may be required to store sampling equipment in a suitable environment as stated in Section 5.0 of the Dallas Lead Quality Assurance Plan. If sampling equipment is stored at the sample bank, issuing this equipment to the sampling teams on a daily basis or as required, will be necessary.

b. Accepting and Logging Samples:

(1) Transfer of sample custody from the sampler to sample bank personnel (any sample bank personnel may accept custody) will normally occur at the sample bank.

(2) Before accepting custody of any samples, sample bank personnel must check all tags and forms for legibility and completeness.

(a) All individual samples must have a completely filled out sample collection tag attached.

(b) Every sample must be identified on the chain-of-custody form.

(c) Each site sampled must have a completely filled out site description form.

(d) Any discrepancy will be corrected before the sample bank personnel will assume custody. If a discrepancy exists that cannot be resolved to the satisfaction of the sample bank personnel, re-sampling, filling out additional tags and forms, and/or re-visiting the site to obtain necessary documentation may be required.

(e) All unused accountable documents must be returned to the sample bank on a daily basis. However, depending upon circumstances such as a sampling team's schedule and route, accountable documents may be retained by the sampling team leader. The sample bank supervisor, however, must be aware of the situation.

(3) After the sampler relinquishes custody and the sample bank personnel assumes custody of the samples, each sample must be logged into the master log book. The master log book should be a suitable ledger with column headings, as shown on Figure 5. Additional headings may be required as the program develops, as such, blank columns should be available in the ledger.

c. Dust Samples:

(1) All sample bank numbers designating dust samples should begin with the capital letter D. The remaining five digits will be numbers beginning with 00001., i.e., the first dust sample logged into the log book will be D00001. The second sample D00002, etc.

Sample Bank No.	Sample Collection Tag No.	Date Collected	Media	Chain-of-Custody Form No.	Site I.D. Code No.	Link No.	Address	Analytical Tag No.	Analytical Laboratory (Initial)	Date Sent to Analytical Laboratory	Chain-of-Custody Form No. to Analytical Laboratory	Remarks
D00001	15031	4 July 82	Dust	05301	RSR-01		109 1st Street	00231	GCA	7 July 82	05490	

Figure 5. Master Log Book Headings.

(2) After assigning and logging the sample bank number, record in the log book the sample collection tag number, link number (No. on computerized preprinted tag), the date the sample was collected, the media (dust), the chain-of-custody form number that the sample appeared on when custody of the sample was assumed by the sample bank, the site I.D. Code and the address where the sample was collected.

(3) Remove the sample collection tag from the sample, attach to the sample an analytical tag, and record the analytical tag number in the log book. Record the sample bank number and the sample description (dust) on the analytical tag. The sample is now ready for shipment or it can be stored until additional samples are logged.

(4) When the samples are prepared for shipment, record in the log book the initials of the analytical laboratory to which the sample is to be sent and the date the sample was shipped.

(5) Fill out a chain-of-custody form (Figure 4), identifying the analytical laboratory to which the samples are to be sent, the sample bank number of each sample in the shipment, the sample description (dust), and any remarks concerning the sample that are appropriate. Record the chain-of-custody form number in the log book.

(6) Remarks concerning the condition of a sample or other pertinent sample information can be beneficial, especially during data interpretation. Record in the log book any information that may be appropriate.

(7) Fill out two analysis data reporting forms (Figure 3, Section 2.0 in Quality Assurance Plan Dallas Lead Study). One form will be included with the sample shipment to the analytical laboratory. Record all available information on this data reporting form except the Sample I.D. Code number and/or site address.

Each sample shipped must be listed on the data reporting form before shipping.

Leave four rows blank on each data reporting form used. These rows will be used for Quality Control samples (See Section 3, Quality Control samples) before sample shipment. The samples can be packaged and shipped as previously identified in the soil sampling protocol.

The second data reporting form will have all available sample information recorded. Include the Site I.D. Code number and/or address on this form.

(8) Make two copies of the second data reporting form, check for legibility. Send one copy to: (RTI)

Mr. Robert M. Lucas
Research Triangle Institute
P.O. Box 12194
Research Triangle Park
North Carolina 27709

and the other copy to: (EMSL-LV)

Dr. Stuart C. Black
U.S. Environmental Protection Agency EMSL-LV
P.O. Box 15027
Las Vegas, NV 89114

(9) The following documentation and forms and their disposition for each sample (i.e., D00001) from collection to the analytical laboratory is as follows:

Form	Disposition
1. Sample Collection Tag	Filed at Sample Bank
2. Site Description Form	Filed at Sample Bank
3. Sampler Chain-of-Custody Form	All copies filled at Sample Bank
4. Master Log Book	Sample Bank
5. Analytical Tag	Analytical Laboratory
6. Chain-of-Custody Form (to Analytical Laboratory)	Original to Analytical Laboratory Copy to Sample Bank
7. Data Reporting Form (with Site I.D. No.)	Original - Sample Bank Copy - RTI Copy - EMSL-LV
8. Data Reporting Form (without Site I.D. No.)	Original - Analytical Laboratory Copy - Sample Bank
9. QC Chain-of-Custody Form	Original - Sample Bank Copy - Originator of QC Sample
10. Shipping Form	Original - Analytical Laboratory Copy - Sample Bank

d. Soil Samples:

(1) All sample bank numbers designating soil samples should begin with the capital letter S. The reamining five digits will be numbers beginning with 00001, i.e., the first soil sample logged into the log book will be S00001, the second soil sample S00002 and etc.

(2) The log book should be arranged in such a manner as to log media in groups. For example, all soils should be logged together, all dust together and etc.

(3) Logging of soil samples will be identical to that described for dust samples. The soil samples, however, must be prepared for analysis by sample bank personnel before they can be shipped to the analytical laboratory. The sample should not be given an analytical tag until after preparation.

(4) After assigning a sample bank number to the soil sample and recording the proper information into the log book, place the soil sample into a properly identified drying container (label the container with the sample bank number). Follow the soil sample preparation procedures as identified in the soil sampling protocol.

(5) If the sample is a split as previously identified in Attachment 1 (those samples collected from row and column grid sampling sites D02, E07, E09, F03, J07, K09, L06, M10, M13, and N05) divide the sample after rotating (mixing) into three equal portions. Package each of the three portions into clean polyethylene containers, attach a completely filled out analysis tag to each sample, assign a sample bank number to the two additional soil samples, place an X, Y and Z designation at the end of the site identification code numbers and log all information into the log book.

(6) All soil samples are to be prepared identically, i.e. drying, sieving and mixing. For those soil samples that are not designated as splits, place one-third (1/3) of the mixed soil in a clean polyethylene container, attach an analysis tag to this sample and record the necessary information into the log book. The remaining two-thirds portion should be repackaged, labeled and identified by a sample bank number and then stored in a secure environment at the sample bank.

(7) Material that is discarded during the sieving procedure must be described, i.e. approximate amount, presence of paint chips and etc. Record this information in the master log book under remarks.

(8) The soil samples can now be prepared for shipment as previously described for dust samples. All documentation and forms used for dust samples are to be used for soil samples.

e. Blood Samples:

(1) Blood samples will not be assigned sample bank numbers. Record and log the link number (number attached to the blood sampling tube) into the sample bank log book. Link numbers will be used to identify blood samples at the analytical laboratory. Analytical tags will not be used. All other sample bank logging documentation and shipping procedure will be similar to that described for dust samples.

(2) Blood samples should be refrigerated while at the sample bank.

(3) Data Reporting forms should not be sent to Dr. Black at EMSL-LV.

f. Vegetation Samples:

(1) All sample bank numbers designating vegetation samples should begin with the capital letter V. The remaining five digits will be numbers beginning with 00001., i.e. the first vegetation sample logged into the log book will be V00001, the second vegetation sample V00002 and etc.

(2) After assigning the sample bank number, record the sample bank number, collection tag number, date collected, media, chain-of-custody form number, and the site I.D. Code number in the log book.

(3) Record the sample bank number in the remarks section on the sample collection tag. Don't remove the sample collection tag from the sample container; store the Vegetation sample as described in the soil sampling protocol.

g. Quality Control Samples:

(1) Quality control samples will be sent to the sample bank accompanied by chain-of-custody forms as shown on Figure 3. These samples will be appropriately identified by sample description and concentration. Additional information may be written in the remarks section of the chain-of-custody form.

(2) These QC samples will be given a sample bank number beginning with the capital letter N. The remaining five digits will be numbers beginning with 00001, i.e. the first Quality Control sample logged into the log book will be N00001, the second N00002 and etc.

(3) These QC samples will be logged in, documented, and shipped identically to procedures previously identified for dust samples.

(4) As previously mentioned, four rows on each data reporting form will be left blank. These rows are for recording data and information concerning analytical laboratory generated QC samples.

The analytical laboratory will fill in the required analytical information on the Data Reporting Form. The sample bank, however, will fill in the sample bank number on the form as follows:

QB0001 will denote a reagent blank
QS0001 will denote a laboratory control standard
QA0001 will denote a split extract

These sample bank numbers will be logged into the log book in the QC section. Also, these sample bank numbers should be logged in numerical order by category, i.e QB0001, QB0002, etc.

(5) The sample bank personnel will generate one sample bank blank after preparing 20 soil samples. The sample bank blank will be DDI water that has come in contact with both the soil sieve and mixing jar.

(6) These sample bank QC samples will be given a sample bank number beginning with the capital letter N followed by a capital letter B. The remaining four digits will be numbers beginning with 0001, i.e. the first sample bank blank logged into the

log book will be NB0001, the second NB0002 and etc. The sample bank blanks will be shipped to the analytical laboratory with the soil samples.

(7) Every soil sample designated as a split by the site identification code number ending with the capital letter X will be sent to

Dr. Ted Meiggs
USEPA
National Enforcement Investigation Center
Building 53, Box 25227
Denver Federal Center
Denver, Colorado 80225

The remaining splits (Y and Z designations) will be sent to the contractor laboratory.

(8) One of the two dust split extracts will be delivered to the sample bank for logging and shipment to Dr. Ted Meiggs.

(9) Duplicate samples designated by the capital letter A at the end of the site identification code will be sent to the same analytical laboratory.

(10) The following is a listing of sample bank identification numbers or link numbers and what they designate.

Number	Designation
D00001	Dust
S00001	Soil, Field Blanks
Link number	Blood
V00001	Vegetation
N00001	QC Samples sent to Sample Bank
QB0001	Reagent Blank
QS0001	Control Standard
QA0001	Split Extract
NB0001	Sample Bank Blanks

APPENDIX B

SOIL AND WIPE SAMPLE ANALYSIS

DALLAS LEAD SMELTER STUDY

Soil and Wipe Sample Analysis

1.0 Analysis of Samples

1.1 Apparatus

1.1.1 Glassware. Class A borosilicate glassware should be used throughout the analysis.

- a. Beakers: 150 ml graduated Pyrex beakers.
- b. Volumetric flasks: 50 ml, 100 ml, 500 ml, and 1000 ml flasks.
- c. Pipettes (to be calibrated at the beginning of the study).
- d. Watch glasses.
- e. Funnels.
- f. Glass stir rods.

1.1.2 Hot plates.

1.1.3 Drying oven.

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1.1.4 Analytical balance capable of weighing to 0.1 mg.

1.1.5 Whatman 42 ashless filter paper, or equivalent.

1.1.6 Atomic Absorption Spectrophotometer equipped with lead hollow cathode lamp.

1.1.7 Acetylene: grade recommended by A.A. manufacturer.

1.1.8 Air: filtered to remove particulate, oil and water.

1.1.9 Mortar and pestle (agate or glass).

1.1.10 100-mesh sieve, U.S. standard or equivalent, with nylon or lead-free wire screen.

1.2 Reagents

1.2.1 Distilled-deionized water (D.I. water).

1.2.2 Concentrated nitric acid (HNO_3), ACS Reagent Grade.

1.2.3 NBS-SRM 928, Lead Nitrate ($Pb(NO_3)_2$) (see Appendix A).

1.2.4 NBS-SRM 1648, Urban Particulate Matter (see Appendix B).

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1.2.5 NBS-SRM 1645, River Sediment (see Appendix C).

1.2.6 EPA Quality Control Samples - Trace Metals - WPI, or equivalent.

1.3 Preparation of Apparatus

All laboratory ware used for extraction, analysis and storage will be prepared in the following manner:

1.3.1 Wash in hot water using a laboratory detergent.

1.3.2 Rinse thoroughly three times with hot tap water to remove all of the detergent.

1.3.3 Immerse in 1:5 (v/v) nitric acid bath and allow to soak for a minimum of four hours.

1.3.4 Remove from the acid bath and rinse three times with deionized water in order to remove all of the acid.

1.3.5 Dry the glassware at 105°C.

1.3.6 Protect all cleaned laboratory wear from dust and fumes by appropriate means (e.g., cover with aluminum foil, put in plastic bag, store in closed containers, etc.)

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1.4 Stock and Working Solution Preparations

1.4.1 Prepare 1:1 (v/v) HNO_3 used for extractions by diluting 500 ml concentrated HNO_3 with D.I. water to 1 liter.

1.4.2 Prepare 1:4 (v/v) HNO_3 used for preparation of standard solutions by diluting 250 ml conc. HNO_3 with D.I. water to 1 liter.

1.4.3 Prepare 1:5 (v/v) HNO_3 used for acid wash by diluting 200 ml of concentrated HNO_3 with D.I. water to 1 liter. Prepare appropriate volume necessary for a polyethylene acid wash tank. Analyze acid wash for lead on every working day and keep record of the daily lead values. Renew solution when a daily analysis shows lead content to be $\geq$ 5 ppm.

1.4.4 Prepare 1:100 (v/v) HNO_3 used for preparation of rinse solutions and extract dilution by diluting 10 ml conc. HNO_3 with D.I. water to 1 liter.

1.4.5 Prepare 1000 µg/ml Pb standard stock solution by dissolving 1.5985 g of NBS-SRM 928, $Pb(NO_3)_2$, in 200 ml of D.I. water contained in a 1000-ml volumetric flask. Acidify with 250 ml conc. HNO_3 and dilute to volume with D.I. water. Record contents and date on label.

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1.4.6 Prepare 100 µg/ml Pb standard solution by diluting 50 ml of 1000 µg/ml Pb standard stock solution to volume in a 500-ml volumetric flask with 1:4 HNO_3. This solution must be prepared fresh on each sample analysis day.

1.4.7 Calibration solution preparation:

Prepare fresh calibration standards of 0.5, 1.0, 2.0, 5.0, 10.0, and 15.0 µg/ml Pb, respectively, on each analysis day by pipetting 0.5, 1.0, 2.0, 5.0, 10.0 and 15.0 mL, respectively, of the 100 µg/ml standard into 100-ml volumetric flasks and bringing to volume with 1:4 HNO_3.

1.5 Calibration of Equipment

1.5.1 Analytical Balance

Weigh a set of class S weights ranging from 1 to 100 g on the analytical balance(s) used during this study at the beginning of the study and repeat every 21 days. If any of the readings do not agree within ±0.2 mg with the stated value, have the balance recalibrated. Record the results of all balance checks in the balance log book. A calibration should also be performed whenever the analytical balance has been moved or has been subjected to rough handling or cleaning.

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1.5.2 Atomic Absorption Spectrophotometer

a. Prepare a set of 6 calibration standards each day when atomic absorption analyses are performed, as described in Section 1.4.7.

b. With the 6 calibration standards ranging in concentration from 0.5 to 15 µg/ml and a blank consisting of 1:4 (v/v) HNO_3 (see 1.4.2), calibrate the atomic absorption spectrophotometer using the procedures recommended by the manufacturer.

c. Plot on graph paper the absorbance data versus concentration and eye-estimate the linear portion of the calibration curve. For the linear region enter into a linear regression algorithm the absorbance versus concentration data to determine the working calibration graph to which subsequent sample absorbances are compared to yield concentration.

1.6 Sample Preparation and Extraction

Two basic types of samples will be collected and analyzed for lead during this study, namely soil samples and dust wipes (cotton swabs which have been used to wipe dust from the interior of houses located in the target area as specified in the House Dust Sampling Protocol).

1.6.1 Soil

The participating laboratory will receive soil sample aliquots consisting of approximately 60 g of soil which has been previously dried, screened, mixed and split. The samples will be contained in 27-ounce whirl-pak polyethylene containers. The soil samples will be prepared for extraction using the following procedures:

a. Hand-mix in its receiving container each soil sample received by tumbling the container 3 to 5 times.

b. Grind with mortar and pestle approximately 5 grams (Note) of sample, sieve through a 100-mesh sieve, and weigh into a tared beaker. Dry for one hour at 105°C, cool, and weigh to the nearest 0.1 mg to determine the exact sample weight. Store the remaining original sample in the original container in a locked room until written permission has been received from EPA stating that the samples can be discarded.

Note 1: After every use, mortar, pestle and sieve shall be cleaned as follows: Wipe off all soil with tissue paper. Rinse three times with D.I. water. Wipe dry with tissue paper. When not in use, mortar, pestle and sieve must be protected from dust and fumes as specified in 1.3.6.

Note 2: The sample size of 5 grams was chosen over 1 gram because the larger sample size will reduce errors due to sample inhomogeneity.

c. Cover the beaker containing the weighed sample with a watch glass and keep it in a secured area until the extraction is performed.

d. To each soil sample add 50 ml of 1:1 HNO_3 and re-cover the beaker with a watch glass.

e. Heat the sample at 95°C and gently boil for 2 hours. Stir sample occasionally with a glass rod. If during this step the volume goes below 25 ml, add more 1:1 HNO_3.

f. Allow the sample to cool, add 10 ml of 1:100 (v/v) HNO_3 and filter through Whatman No. 42 filter paper (or equivalent) into a 100-ml volumetric flask. Rinse the watch glass, glass rod, beaker and filter paper with two 10-ml portions of 1:100 HNO_3 and collect rinsings in the volumetric flask. Bring to volume with 1:100 HNO_3. The sample is now ready for analysis.

1.6.2 Dust Wipe Samples

It is anticipated that each dust wipe sample will consist of two individual swabs.

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a. With teflon-coated tweezers, transfer the dust wipe sample swabs to a beaker for extraction. Dust wipe samples are not weighed as the analytical results are reported as total micrograms of lead per square centimeter wiped.

Note: Make sure that both swabs which make up one individual sample are placed in the same beaker.

b. Cover the beaker containing the dust wipe sample with a watch glass and keep it in a secured area until the extraction is performed.

c. To each sample consisting of 2 swabs, add 30 ml of 1:1 HNO_3 and cover the beaker with a watch glass.

d. Heat the sample at 95°C and gently boil for 2 hours. Stir sample occasionally with a glass rod. If during this step the volume goes below 15 ml, add more 1:1 HNO_3.

e. Allow the sample to cool, add 10 ml of 1:100 (v/v) HNO_3 and filter through Whatman No. 42 filter paper (or equivalent) into a 50-ml volumetric flask. Rinse the watch glass, glass rod, beaker and filter paper with two 10-ml portions of 1:100 HNO_3 and collect rinsings in the volumetric flask. Bring to volume with 1:100 HNO_3. The sample is now ready for analysis.

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1.7 Atomic Absorption Analysis for Lead

1.7.1 Install lead hollow cathode lamp. Set wavelength of the monochromator at 283.3 nm. Set instrument operating conditions as recommended by the manufacturer. Background correction is required for these analyses.

1.7.2 At the beginning of each analysis set, but at least once per shift, calibrate the instrument as described in 1.5.2. Immediately following calibration, aspirate a known control sample such as the EPA QA Water Samples - WPI, independent of the calibration standards. Refer to Section 2.1.2 for details on use.

1.7.3 Aspirate sample extracts into flame. Take the mean of three integrations per sample and record this value (equilibrium absorbance) and the sample number on the strip chart tracing (or equivalent). Dilute 1:10 all samples, that exceed the highest working standard, with 1:4 HNO_3 so that their absorbances are within the linear working range. If the 1:10 dilution is not sufficient then dilute the original extract 1:100 with 1:4 HNO_3.

1.7.4 After each 10 samples aspirate one calibration standard of 10 µg/ml. If the standard result deviates by more than 5 percent from the value predicted by the calibration curve, recalibrate and repeat the previous 10 analyses.

1.7.5 After every 20 samples aspirate the known control sample as in 1.7.2.

1.8 Data Reduction and Reporting

1.8.1 Soil sample analytical results are to be expressed as micrograms of lead per gram of soil (dry weight) and are calculated using the following equation:

$$\mu g\ Pb/gm\ (dry\ weight) = ABC/D$$

where A = µg/ml of Pb in sample extract
B = Final volume of sample extract (100 ml)
C = Dilution factor (10 or 100), if necessary
D = Sample weight in grams (dry weight)

The minimum reportable level is 4 µg Pb/gm based on twice an instrumental detection limit for lead of 0.1 µg Pb/ml.

1.8.2 Results from dust wipe samples are to be expressed in micrograms of lead per square centimeter of area wiped and are calculated using the following equation:

$$\mu g\ Pb/cm^2 = ABC/D$$

where A = µg/ml of Pb in sample extract
B = Final volume of sample extract (50 ml)

C = Dilution factor (10 or 100), if necessary

D = 100 cm^2

The minimum reportable level is 0.1 μg Pb/cm^2 based on twice an instrumental detection limit for lead of 0.1 μg Pb/ml.

1.8.3 The results are reported for sets of 20 samples to the designated organizations on Sample Analysis Report Forms (Figures 1 and 2) together with all required QC data which are listed for each set of 20 samples on a separate form (Figures 3 and 4).

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Date: 6/16/82

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Soil Sample Analysis Report

Report to: ______________________________ Date Reported: ______________

Distribution: ____________________________

____

____

Laboratory Name: ____________________________________

Date Sample Received	Field Sample ID	Lab Sample ID	Date Extraction Performed, Analyst Initials	Date Analysis Completed, Analyst Initials	Result µg Pb/gm Soil

____
Laboratory Supervisor

Figure 1

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DALLAS LEAD SMELTER STUDY

Housedust Sample Analysis Report

Report to: ______________________ Date Reported: __________

Distribution: ______________________

____

____

Laboratory Name: ______________________

Date Sample Received	Field Sample ID	Lab Sample ID	Date Extraction Performed, Analyst Initials	Date Analysis Completed, Analyst Initials	Result µg Pb/cm²

____
Laboratory Supervisor

Figure 2

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SOIL SAMPLE QUALITY ASSURANCE

Report to: ____________________ Date Reported: ____________________

Distribution: ____________________ QC Report # ____________________

____________________ Sample #'s: (Sequence as analyzed)

____________________ ____________________

____

Laboratory name: ____________________

Laboratory supervisor: ____________________ Analyst(s) initials: __________

---

1. Reagent blank: µg/ml ____________________
(see 2.1.1)

---

2. Field blank: µg/ml ____________________
(see 2.1.3)

Sample # ____________________

---

3. Calibration control sample: µg/ml:
(see 2.1.2)

Source: ____________________

Found ____________________

True ____________________

% recovery ____________________

---

4. Spike extract analysis: µg/ml

Sample # ____________________
(see 2.1.5)

Sample extract result ____________________

Spike extract result ____________________

Spike added ____________________

% recovery ____________________

---

5. Residue re-extraction analysis: µg/gm
(see 2.1.6)

Sample # ____________________

Original extraction ____________________

Residue extraction ____________________

% originally extracted ____________________

Figure 3. (continued)

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6. Total recoverable analysis: µg/gm (see 2.1.7)

Sample # ____________________

Original result ________________

"Total" method result ________________

7. Laboratory control sample analysis: µg/gm (see 2.1.8)

Source: NBS SRM 1645, River Sediment

Found ________________

True ________________

% recovery ________________

8. Split extract analysis: µg/gm

Sample # __________
(see 2.1.9)

Original extract result ________________

Split extract result ________________

9. Spiked sample analysis: µg/gm

Sample # __________
(see 2.1.10)

Spiked sample result ________________

Unspiked sample result ________________

Spike added ________________

% recovery ________________

Figure 3

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DUST WIPE SAMPLE QUALITY ASSURANCE

Report to: ____________________
Distribution: ____________________
____
____

Date Reported: ____________________
QC Report # ____________________
Sample #'s: (Sequence as analyzed)
____
____

Laboratory name: ____________________
Laboratory supervisor: ____________________ Analyst(s) initials: ________

---

1. Reagent blank: µg/ml ____________________
(see 2.2.1)

---

2. Field blank: µg/ml^2 ____________________
(see 2.2.3)
Sample # ____________________

---

3. Calibration control sample: µg/ml:
(see 2.2.2)
Source: ____________________
Found ____________________
True ____________________
% Recovery ____________________

---

4. Spike extract analysis: µg/ml
(see 2.2.4)
Sample # ____________________
Sample extract result ____________________
Spike extract result ____________________
Spike added ____________________
% Recovery ____________________

---

5. Laboratory control sample analysis: weight % Pb
(see 2.2.5)
Source: NBS SRM 1648, Urban Particulate
Found ____________________
True ____________________
% Recovery ____________________

---

6. Split extract analysis: µg/cm^2
Sample # ____________________
(see 2.2.6)
Original extract result ____________________
Split extract result ____________________

Figure 4

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2.0 Quality Control Procedures

The following quality control analyses will be performed at the stated frequencies:

Soil:

a. one reagent blank (one per 20)
b. one calibration control sample (one per 20)
c. one field blank (one per 20)
d. one duplicate sample (blind) (one per 20)
e. one spiked extract (one per 20)
f. one reextraction of sample residue (one per 20)*
g. one "total recoverable" analysis (one per 10)*
h. one laboratory control sample (one per 20)*
i. one split extract analysis (one per 20)
j. one spiked sample (one per 20)*

Dust Wipes:

a. one reagent blank (one per 20)
b. one calibration control sample (one per 20)
c. one field blank (one per 20)
d. one spiked extract (one per 20)
e. one laboratory control sample (one per 20)*
f. one split extract (one per 20)

---

*The frequency of these analyses may be decreased during the study.

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In order to minimize bias the QC samples are intermixed with the field samples and the analysis sequence following calibration and analysis of the calibration control sample (b) will be randomly changed. The laboratory shall order the analysis sequence for every set of 20 samples as follows for soil (and similarly for dust), and then refer to a random number table to determine where to start the analysis sequence.

1. Perform calibration and calibration control samples analysis. After every tenth sample, reanalyze a calibration standard (see Section 1.7.4).

2. Order 20 field samples (X_1 to X_{20}) and corresponding QC samples as follows: X_1, X_2, X_3, a, X_4, X_5, X_6, e, X_7, X_8, X_9, g, X_{10}, X_{11}, X_{12}, f, X_{13}, X_{14}, X_{15}, h, X_{16}, X_{17}, X_{18}, i, X_{19}, X_{20}, g, j, . . . continue with b and next sequence
   where: X_1 . . . X_{20} are samples 1-20 and
   where: a, e, g, f, h, i, j, and b are the QC analyses specified above. [QC samples c and d are not included since they are to be treated as samples.]

3. Refer to a random number table to determine where to start the analysis sequence (X_1 or e or etc.).

4. For dust wipe analyses a similar type of sequence is to be used. X_1 . . . X_5, a, X_6 . . . X_{10}, d, X_{11} . . . X_{15}, e, X_{16} . . . X_{20}, f . . . continue with b and next sequence.

2.1 Soil Analysis Quality Control

2.1.1 Reagent Blank Analysis

a. A reagent blank will be processed and analyzed with each set of 20 samples, on each day that soil extractions are performed, to establish the background lead level.

b. The reagent blank analysis will be performed as outlined in Section 1.6.1 for the soil samples except that no soil sample is added. Results are to be reported on the Soil Sample Quality Assurance Form. If the concentration of lead in the reagent blank exceeds 0.2 µg/ml (one-half the lowest calibration standard) then sources of contamination should be identified and eliminated, and EMSL-LV should be contacted immediately for assistance.

2.1.2 Calibration Control Sample

a. In order to verify the instrument calibration, a known control sample, such as the EPA Quality Assurance Water Samples - Trace Metals - WPI (prepared by EMSL-CIN), will be analyzed immediately

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following calibration and after every 20 samples (see Section 1.7.5). These standards will be provided by EMSL-LV in a concentrated form. The participating laboratory will dilute the concentrate to fall within the calibration range of the AAS for lead.

b. Calculate the percent recovery, using the certified lead concentration of the control sample, as diluted, and the analyzed concentration, according to the following equation:

$$\% \text{ recovery} = A/B \times 100$$

where: A = analyzed concentration of lead

B = certified concentration of lead (as diluted).

Results are to be reported on the Soil Sample Quality Assurance Form.

c. Maintain control charts on the percent recovery and establish through separative analyses the 99 and 95% confidence limits (control and warning).

d. If the percent recovery is outside of the acceptable range of 95 to 105% or outside the 95% confidence limits, whichever is less, then corrective action such as recalibration and preparation of fresh working and/or stock lead solutions must be taken. EMSL-LV will be available for assistance if required.

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2.1.3 Field Blank Analysis

For every 20 soil samples collected in the field one soil container filled with DI water will be sent to the Sample Bank and then forwarded to the participating laboratory. The entire contents of the container will be transferred to a beaker, carefully evaporated to near dryness and then processed as specified in Section 1.6.1 for soil samples. Results are to be reported on the Soil Sample Quality Assurance Form.

2.1.4 Duplicate Sample Analysis (Blind)

One in 20 soil samples sent to the Sample Bank will be split and forwarded as a blind duplicate to the participating laboratory.

2.1.5 Spiked Extract Analysis

For each 20 soil samples, one sample extract is spiked with lead nitrate to determine the effect of the extract matrix on the accuracy of the analysis procedure.

a. A laboratory-predetermined soil sample is extracted and analyzed as previously described (Section 1.6.1).

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b. To an aliquot of this sample extract is added an amount of lead (as lead nitrate) approximately equal to the lead content determined for this extract but greater than 10 x the instrumental detection limit. This is best accomplished with 1000 ug/ml Pb standard stock solution using a calibrated micropipet such that the extract volume is not appreciably changed.

c. Directly analyze the spiked extract as described in Section 1.7.

d. Calculate the percent recovery of the spike using the following equation:

$$\% \text{ recovery} = (A-B)/C \times 100$$

where: A = analyzed total lead content of the spiked extract
B = analyzed lead content of the unspiked extract
C = lead content of spike.

Results are to be reported on the Soil Sample Quality Assurance Form. If the percent recovery is outside of the acceptable range of 90 to 110% then repeat the spiked extract analysis on two additional sample extracts from the same set of 20 samples. If the result from either of the additional spiked extracts is outside the acceptable range then use the method of additions for analysis of all 20 samples as described in EPA-600/4-79-020, March 1979 (Appendix 4).

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2.1.6 Reextraction of Sample Residue

a. For each 20 samples, one sample residue consisting of the filter paper and the undissolved soil portion will be extracted and analyzed according to the procedures set forth in Section 1.6.1.

b. Calculate the percent of lead extracted by the first extraction based on the total of two extractions as follows:

$$\% \text{ extracted} = \frac{A}{A + B} \times 100$$

where: A = ug Pb/gm from first extraction

B = ug Pb/gm from second extraction (based on original sample weight).

c. Report results on the Soil Sample Quality Assurance Form.

2.1.7 "Total Recoverable" Analysis

For each 10 samples, a "total recoverable" analysis must be performed. The extraction method chosen for this step has been demonstrated to give equivalent results for a variety of soil types when compared to known "total" methods. However, it is still necessary to closely monitor the extraction efficiency

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throughout this study. Once sufficient data have been gathered, reevaluation of the frequency of the "total recoverable" analysis may be considered. It is expected that the results from the "total recoverable" method detailed below will agree within 10% with those generated using the method detailed in Section 1.6.1. If after the analysis of 200 samples this agreement can be verified then the frequency of the "total recoverable" analyses might be lowered.

Note: The more rigorous HF-$HClO_4$-HNO_3 method which is widely used for total metals analysis in soil was considered but not chosen. This method is overly cumbersome, time-consuming and requires special equipment, and it was felt that any additional information that may be gained using the HF method would not warrant the additional effort.

DIGESTION PROCEDURE

Weigh and transfer to a 150 ml beaker a 5.0-gram portion of sample which has been prepared as discussed in Section 1.6.1. Add 25 ml of 1:1 HNO_3 and cover with a watch glass. Heat the sample at 95°C and reflux to near dryness. Allow the sample to cool, add 10 ml of conc. HNO_3 and again reflux to near dryness. After the second reflux step has been completed and the sample has cooled, add 5 ml of 1:1 HNO_3 and 3 ml of 30% hydrogen peroxide

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(H_2O_2). Return the beaker to the hot plate for warming to start the peroxide reaction. Care must be taken with the start of effervescence that losses do not occur or the reaction is not too vigorous. Heat until effervescence subsides and cool the beaker. Continue the addition of 30% H_2O_2 in 1-ml aliquots with warming until the effervescence is minimal or the general sample appearance is unchanged. Do not add more than a total of 10 ml 30% H_2O_2.

Add 5 ml of 1:1 HNO_3, return the covered beaker to the hot plate and reflux for an additional 10 min. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 100 ml with 1:4 nitric acid. The sample is now ready for analysis according to Section 1.7.

Results are to be reported in ug Pb/gm on the Soil Sample Quality Assurance Form. If the "total recoverable" analysis results differ by more than 10% from the results obtained according to 1.6 and 1.7, EMSL-LV should be contacted immediately for assistance.

2.1.8 Laboratory Control Sample Analysis.

a. One NBS-SRM 1645, River Sediment, sample will be analyzed for each 20 field soil samples, as an independent certified control sample.

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b. The samples are to be prepared and analyzed as detailed in Appendix C and in Section 1.6.1 and 1.7.

c. Report on the Soil Sample Quality Assurance Form the results and the percent recovery of NBS certified concentration as calculated below:

$$\% \text{ recovery} = B/A \times 100$$

where: A = NBS SRM certified lead concentration
B = analyzed lead concentration

d. Maintain control charts on the percent recovery and establish through repetitive analyses the 99 and 95% confidence limits (control and warning).

e. If the percent recovery is outside of the acceptable range of 90 to 110%, or outside the 95% confidence limits, whichever is less, the analysis will be repeated. If the percent recovery is outside the acceptable range with the repeat sample, a thorough check of the extraction and analysis procedures should be conducted before proceeding with additional field sample analyses. EMSL-LV should be contacted immediately for assistance.

2.1.9 Split Extract Analysis

Intralaboratory precision for the analysis only will be estimated from the results of two replicate aliquots (split extracts) from every twentieth sample:

a. For each 20 soil sample extracts, split the extract of one sample into two portions and analyze each by atomic absorption.

b. Report the results of the split extract analysis on the Soil Sample Quality Assurance Form.

2.1.10 Spiked Sample Analysis

In order to monitor potential lead losses during extraction, a spiked sample analysis will be performed on a duplicate portion of one sample for each 20 field samples. Once sufficient data have been gathered, the frequency of the spiked sample analysis may be reconsidered.

a. For each 20 samples, a duplicate portion of one sample is spiked with an amount of lead, as lead nitrate solution, about equal to the lead concentration of the sample as analyzed, and processed as specified in 1.6 and 1.7.

b. Calculate the percent recovery of the spike as follows:

$$\% \text{ recovery} = \frac{A - B}{C} \times 100$$

where: A = ug Pb/gm determined in spiked sample

B = ug Pb/gm determined in unspiked sample

C = ug Pb/gm added.

Results are to be reported on the Soil Sample Quality Assurance Form.

c. Maintain control charts on the percent recovery and establish through repetitive analyses the 99 and 95% confidence limits (control and warning).

d. No limits can be set at this time due to unknown sample homogeneity. However, if the percent recovery is outside of the 95% control limits, repeat the spiked sample analysis on a second sample from the same set of 20 samples. If this sample recovery is also outside of the 95% control limits, contact EMSL-LV immediately for assistance.

2.2 Dust Wipe Analysis Quality Control.

2.2.1 Reagent Blank Analysis.

a. A reagent blank will be processed and analyzed with each set of 20 samples on each day that dust wipe extractions are performed to establish the background lead level.

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b. The reagent blank analysis will be performed as outlined in Section 1.6.2 for the dust wipe samples except that no swabs are added. Results are to be reported on the Dust Wipe Sample Quality Assurance Form. If the concentration of lead in the reagent blank exceeds 0.2 ug/ml (one-half the lowest calibration standard), then sources of contamination should be identified and eliminated, and EMSL-LV should be contacted immediately for assistance.

2.2.2 Calibration Control Sample.

a. In order to verify the instrument calibration, a known control sample, such as the EPA Quality Assurance Water Samples-Trace Metals-WPI (prepared by EMSL-CIN), will be analyzed immediately following calibration and after every 20 samples (see Section 1.7.5). These standards will be provided by EMSL-LV in a concentrated form. The participating laboratory will dilute the concentrates to fall within the calibration range of the AAS for lead.

b. Calculate the percent recovery, using the certified lead concentration of the control sample (taking into consideration the dilution) and the analyzed concentration, according to the following equation:

$$\% \text{ recovery} = A/B \times 100$$

where: A = analyzed concentration of lead
B = certified concentration of lead (as diluted).

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c. Maintain control charts on the percent recovery and establish through repetitive analyses the 99 and 95% confidence limits (control and warning).

Results are to be reported on the Dust Wipe Sample Quality Assurance Form. If the percent recovery is outside of the acceptable range of 95 to 105% or outside the 95% confidence limits, whichever is less, then corrective action, such as recalibration and preparation of fresh working and/or stock lead solutions must be taken. EMSL-LV will be available for assistance if required.

2.2.3 Field Blank Analysis.

For every 20 dust wipe samples collected in the field, one field blank (consisting of two swabs) will be delivered to the laboratory. The field blank sample will be processed and analyzed as specified in Section 1.6.2. Results are to be reported on the Dust Wipe Sample Quality Assurance Form. The field blanks will be clearly identified as such on the labels and accompanying forms.

2.2.4 Spiked Extract Analysis.

For each 20 field dust samples, one sample extract is spiked as follows with lead nitrate to determine the effect of the extract matrix on the accuracy of the analysis procedure.

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a. A laboratory-predetermined dust wipe sample is extracted and analyzed as previously described (Section 1.3).

b. To an aliquot of this sample extract is added an amount of lead (as lead nitrate) approximately equal to the lead content determined for this extract, but greater than 10 x the instrumental detection limit. This is best accomplished with 1000 ug/ml Pb standard stock solution using a calibrated micropipet such that the extract volume is not appreciably changed.

c. Directly analyze the spiked extract as described in Section 1.7.

d. Calculate the percent recovery of the spike using the following equation:

$$\% \text{ recovery} = (A-B)/C \times 100$$

where: A = analyzed total lead content of the spiked extract
B = analyzed lead content of the unspiked extract
C = lead content of spike.

Results are to be reported on the Dust Wipe Sample Quality Assurance Form. If the percent recovery is outside of the acceptable range of 90-110%, then repeat the spiked extract analysis on two additional sample extracts from the same set of 20 samples. If the result from either of the additional spiked

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extracts is outside the acceptable range, then use the method of additions for analysis of all 20 samples as described in EPA-600/4-79-020, March 1979 (Appendix D).

2.2.5 Laboratory Control Sample Analysis.

For each 20 field dust wipe samples, one laboratory control sample (NBS SRM 1648, Urban Particulate Matter) will be analyzed. The laboratory control sample is prepared in the laboratory as follows:

a. Dry the material as specified in Appendix B. Weigh 0.1 gram (to the nearest 0.1 mg) of NBS SRM 1648, Urban Particulate Matter, into a tared beaker and then carefully add two freshly unwrapped dust swabs.

b. Analyze the control sample as described in Section 1.6.2.

c. Report on the Dust Wipe Quality Assurance Form the results as % Pb of the Urban Particulate Matter, and calculate the percent recovery based on the NBS certified concentration as follows:

$$\% \text{ recovery} = B/A \times 100$$

where: A = NBS SRM certified concentration
B = analyzed lead concentration.

d. Maintain control charts on the percent recovery and establish through repetitive analyses the 99 and 95% confidence limits (control and warning).

e. If the percent recovery is outside of the acceptable range of 95 to 105%, or outside the 95% confidence limits, whichever is less, prepare a fresh control sample and repeat the extraction and analysis procedures. If on the repeat sample the percent recovery is outside of the acceptable range of 95 to 105%, or the 95% confidence limits, whichever is less, a thorough check of the extraction and analysis procedures should be conducted before proceeding with additional field samples. EMSL-LV should be contacted immediately for assistance.

2.2.6 Split Extract Analysis

Both the overall measurement precision and the intralaboratory precision will be estimated from the split extract analysis.

a. For each 20 dust wipe sample extracts, split the extract of one sample into three portions.

b. Send one fraction to the referee laboratory, NEIC, with all proper identification and chain-of-custody information.

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c. Analyze the other two portions in the set sequence by atomic absorption.

d. Report the results of the split extract analysis on the Dust Sample Quality Assurance Form.

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7.0 COST ESTIMATE

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27 July 1982

Page 1 of 7 Pages

COST ESTIMATE - SAMPLE BANK TASK

Contract No. 68-02-3168, Work Assignment No. 80, Technical Service Area 3
(GCA 1-619-080)

Group Scientist/Engineer	40 hours @ $20.22/hr(2)	$ 809
Staff Scientist/Engineer	80 hours @ $13.53/hr	1,082
Senior Scientist/Engineer	280 hours @ $11.68/hr	3,270
Scientist/Engineer	280 hours @ $ 9.67/hr	2,708
Junior Scientist/Engineer	280 hours @ $ 7.82/hr	2,190
Senior Technician	280 hours @ $ 7.57/hr	2,120
Technical Illustrator	60 hours @ $ 7.56/hr	454
Technical Typist	120 hours @ $ 6.27/hr	752
Total Direct Labor	1,420 hours(1)	$13,385
Salary Related Cost(3) (32.5%)		4,350
Subtotal		17,735
Engineering Overhead(3) (88.0%)		15,607
Travel (see Page 5)		18,589
Purchased Material (see Page 6)		998
Subtotal		52,929
General & Administrative Expense(3) (3.7%)		1,958
Total Estimated Cost		54,887
Fixed Fee(4)		3,125
TOTAL SAMPLE BANK COST		$58,012

7.0 COST ESTIMATE

GCA/TECHNOLOGY DIVISION
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27 July 1982

Page 2 of 7 Pages

COST ESTIMATE - ANALYTICAL TASK

Contract No. 68-02-3168, Work Assignment No. 80, Technical Service Area 3
(GCA 1-619-080)

Group Scientist/Engineer	80 hours @ $20.22/hr(2)	$ 1,618
Staff Scientist/Engineer	100 hours @ $13.53/hr	1,353
Senior Scientist/Engineer	80 hours @ $11.68/hr	934
Scientist/Engineer	340 hours @ $ 9.67/hr	3,288
Junior Scientist/Engineer	360 hours @ $ 7.82/hr	2,815
Senior Technician	980 hours @ $ 7.57/hr	7,419
Technical Illustrator	100 hours @ $ 7.56/hr	756
Technical Typist	200 hours @ $ 6.27/hr	1,254
Total Direct Labor	2,240 hours(1)	$19,437
Salary Related Cost(3) (32.5%)		6,317
Subtotal		25,754
Engineering Overhead(3) (88.0%)		22,664
Purchased Material (see Page 7)		9,066
Subtotal		57,484
General & Administrative Expense(3) (3.7%)		2,127
Total Estimated Cost		59,611
Fixed Fee(4)		4,889
TOTAL ANALYTICAL COST		$64,500

7.0 COST ESTIMATE

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Page 3 of 7 Pages

COST ESTIMATE - ANALYSIS OF DALLAS SOIL SAMPLES(5)

Contract No. 68-02-3168, Work Assignment No. 80, Technical Service Area 3
(GCA 1-619-080)

Group Scientist/Engineer	120 hours @ \$20.22/hr(2)	\$ 2,427
Staff Scientist/Engineer	180 hours @ \$13.53/hr	2,435
Senior Scientist/Engineer	360 hours @ \$11.68/hr	4,204
Scientist/Engineer	620 hours @ \$ 9.67/hr	5,996
Junior Scientist/Engineer	640 hours @ \$ 7.82/hr	5,005
Senior Technician	1,260 hours @ \$ 7.57/hr	9,539
Technical Illustrator	160 hours @ \$ 7.56/hr	1,210
Technical Typist	320 hours @ \$ 6.27/hr	2,006
Total Direct Labor	3,660 hours(1)	\$32,822
Salary Related Cost(3) (32.5%)		10,667
Subtotal		43,489
Engineering Overhead(3) (88.0%)		38,271
Travel (see Page 5)		18,589
Purchased Material (see Pages 6 and 7)		10,064
Subtotal		110,413
General & Administrative Expense(3) (3.7%)		4,085
Total Estimated Cost		114,498
Fixed Fee(4)		8,014
TOTAL WORK ASSIGNMENT COST		\$122,512

27 July 1982

NOTES PERTAINING TO PAGES 1, 2, and 3:

(1) The pertinent technical man-hours are: 1,240 hours (Page 1 - Sample Bank), 1,940 hours (Page 2 - Analytical) for a combined total of 3,180 hours in contrast to the total Work Assignment Government estimate of 2,145 hours.

(2) Use of direct labor bidding rate schedule applicable to 1982.

(3) Use of GCA/FY 1982 provisional indirect rates requested on 14 January 1982.

(4) In accordance with Article VIII, Paragraphs C.1 and C.2 of Option No. 2 to the basic contract, the fees employed herein are calculated at $2.520 per technical man-hour.

(5) This cost estimate pertinent to Work Assignment No. 80 has been obtained by summary of the individual cost elements on Pages 1 and 2. Subsequent monthly financial cost and expenditure breakdowns by activity will necessarily be estimated values (see Paragraph VI.B of Work Assignment No. 80).

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27 July 1982

DETAILED TRAVEL ESTIMATE BY TASK
(GCA 1-619-080)

A. SAMPLE BANK

1. Boston, Mass. - Dallas, Texas

1 Trip | 3 Days/Trip | 2 Man/Trip

Air Fare	- 2 Trip @ $531/Trip	$ 1,062
Per Diem	- 6 Days @ $ 60/Day	360
Car Rental	- 3 Days @ $ 45/Day	135
Local Travel	- 2 Trip @ $ 30/Trip	60
		$ 1,617

2. Boston, Mass. - Dallas, Texas

1 Trip | 28 Days/Trip | 3 Man/Trip

Air Fare	- 3 Trip @ $531/Trip	$ 1,593
Per Diem	- 84 Days @ $ 60/Day	5,040
Car Rental	- 28 Days @ $ 45/Day	1,260
Local Travel	- 3 Trip @ $ 30/Trip	90
		$ 7,983

3. Boston, Mass. - Dallas, Texas

1 Trip | 21 Days/Trip | 4 Man/Trip

Air Fare	- 4 Trip @ $ 531/Trip	$ 2,124
Per Diem	- 84 Days @ $ 60/Day	5,040
Car Rental	- 21 Days @ $ 45/Day	945
Local Travel	- 4 Trip @ $ 30/Trip	120
		$ 8,229

4. Boston, Mass. - Washington, D.C.

1 Trip | 2 Days/Trip | 2 Man/Trip

Air Fare	- 2 Trip @ $185/Trip	$ 370
Per Diem	- 4 Days @ $ 60/Day	240
Car Rental	- 2 Days @ $ 45/Day	90
Local Travel	- 2 Trip @ $ 30/Trip	60
		$ 760

TOTAL SAMPLE BANK TRAVEL $18,589

TOTAL WORK ASSIGNMENT TRAVEL $18,589

PURCHASED MATERIAL BY TASK
(GCA 1-619-080)

A. SAMPLE BANK

1.	Polyvinyl gloves, X-lg., Curtin Matheson, Woburn, MA - 10 pkgs @ $13/pkg	$ 130
2.	Kim wipes, Curtin Matheson - 1 case @ $80/case.	80
3.	Parafilm, clear, grade M, Curtin Matheson - 1 case @ $100/cs	100
4.	Spatula, metal, Curtin Matheson - 5 each @ $4/ea.	20
5.	Wax pencils, Curtin Matheson - 1 package @ $12/ea	12
6.	Alconox, Curtin Matheson - 2 cartons @ $8/ea	16
7.	Rubber gloves, Curtin Matheson - 4 prs @ $5/ea	20
8.	Mortar & pestle, Curtin Matheson - 6 each @ $15/ea.	90
9.	Polythylen wash bottles, Curtin Matheson - 2 packages @ $5/ea	10
10.	Laboratory timer, Curtin Matheson - 1 each @ $20/ea	20
11.	Miscellaneous purchased material including foil, office supplies, etc.	100
12.	Roalox mill jars, Model 774, Curtin Matheson - 4 each @ $100/ea	400
	TOTAL SAMPLE BANK PURCHASED MATERIAL	$ 998

B. ANALYTICAL

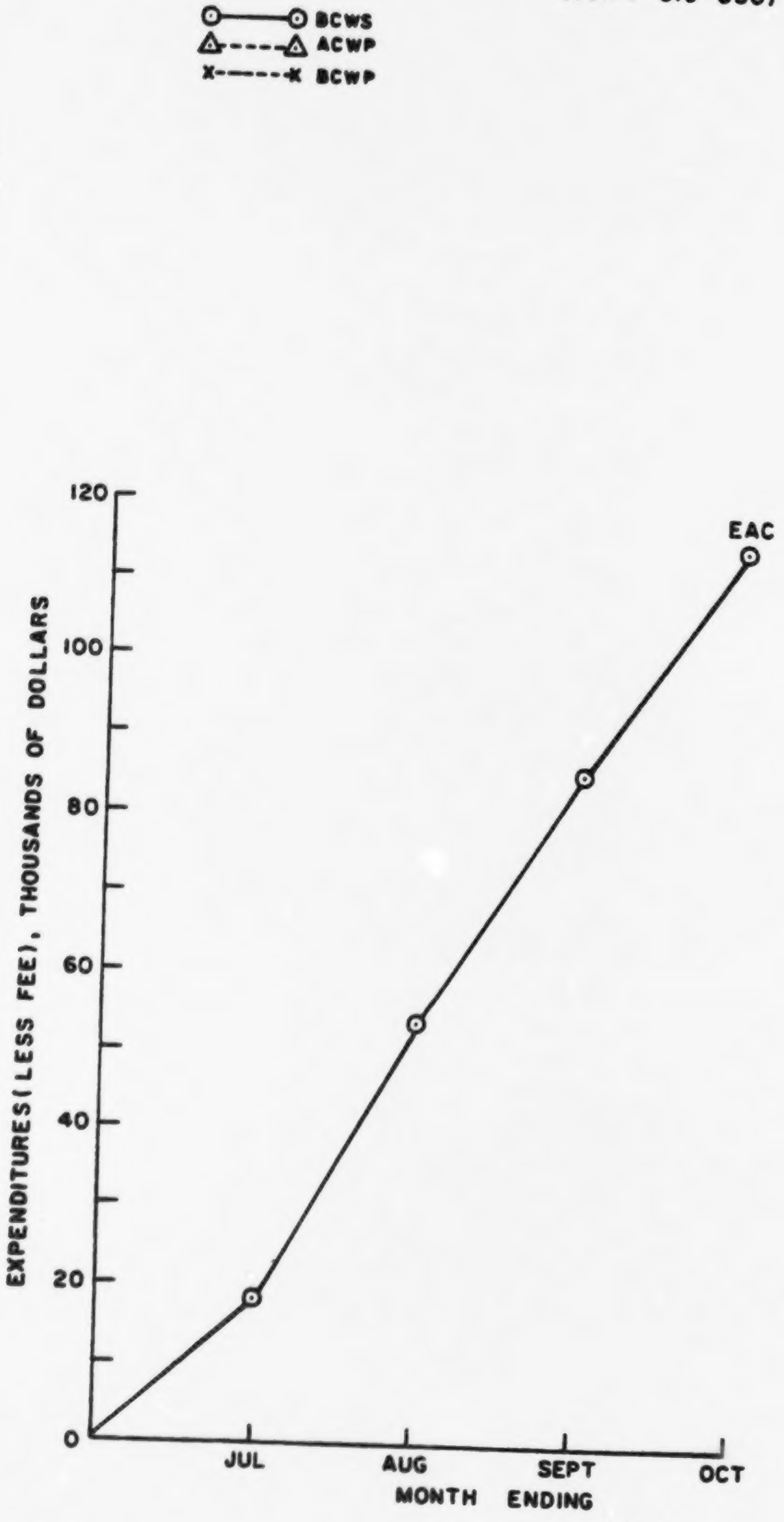
1.	Acetylene, Northeast Cryogenics, Newtonville, MA - 5 tanks @ $17/ea	$ 85
2.	Beakers, 150 ml, Curtin Matheson - 13 cases @ $47.04/ea. . .	612
3.	Volumetrics, 100 ml, Curtin Matheson - 25 cases @ $102.96/ea	2,574

PURCHASED MATERIAL BY TASK
(GCA 1-619-080)

4.	Speedy vaps, 75mm, Fisher Scientific, Medford, MA - 1 case @ $145.75/ea	$ 146
5.	Funnels, glass, Curtin Matheson - 1 case @ $100.80/ea . .	101
6.	Glass stir rods, Curtin Matheson - 2 pkgs @ $14.67/pkg. .	29
7.	Whatman filter paper, #42, Curtin Matheson - 10 packages @ $9/ea	90
8.	Mortar & Pestles, Porcelain, Curtin Matheson - 12 sets @ $28/ea	336
9.	Brass sieves, 100 mesh, Curtin Matheson - 12 each @ $30.25/ea	363
10.	Funnel rack, Arthur H. Thomas, Philadelphia, PA - 10 each @ $16.25/ea	163
11.	Instra analyzed nitric acid, 2.5 1, Curtin Matheson - 15 cases @ $111.60/ea	1,674
12.	Petri dishes, bottoms only, Curtin Matheson - 1 case @ $97.92/ea	98
13.	4 ox. Nalgenes, Curtin Matheson - 70 cases @ $36.72/ea. .	2,570
14.	Hollow cathode Pb lamp, Perkin Elmer, Wellesley, MA - 1 each @ $225/ea	225
	TOTAL ANALYTICAL PURCHASED MATERIAL	$9,066

TOTAL WORK ASSIGNMENT PURCHASED MATERIAL $10,064

CUMULATIVE PERFORMANCE
CONTRACT NO. 68-02-3168, TECHNICAL SERVICE AREA 3
WORK ASSIGNMENT NO. 80
(GCA 1-619-080)



APPENDIX I

DALLAS LEAD STUDY LESSONS LEARNED

FIELD SAMPLE BANK

The management of the Dallas Sample Bank encompassed two major tasks. The first task involved the receipt, logging, and disbursement to the appropriate laboratory of all program samples. The second dealt with the homogenization and handling of the soil samples prior to shipment.

The most significant influence adversely affecting efficient sample bank operation was the sizeable accumulation of samples prior to the initiation of bank management. Future programs of this nature would benefit by the firm establishment of the field sample bank coincident to the sampling effort.

The following recommendations are offered for utilization in future similar programs:

- Onsite training of sampling crews and sample bank personnel in actual "hands on" procedures related to the sampling crew/sample bank interface.
- Designation of Field Sample Bank as the focal point of all field operations. This should include the dissemination of all sample control documents and pertinent sampling supplies.
- Complete standardization of document and handling procedures for all samples matrices. Site and sample identification should probably not be included as information transmitted to the analytical laboratory in order to preclude biasing of results. All blood samples transmitted to the analytical laboratory were accompanied by site identification codes.
- Level of effort should be maximized at the onset of the program and gradually decreased as the program progresses. As indicated in Figure 3-1 in Section 3, training of supplementary personnel occurred at the latter stage of the project. This effort would have been more efficient and cost effective if carried out at the onset of the program.
- Preserialization of documents related to sample identification and control.

ANALYSIS AND DATA HANDLING

Analysis of NBS SRM 1645, river sediment, was accomplished by utilizing -g and 1-g aliquots. It was recognized that greater analytical difficulties were encountered during the analysis of 5-g portions. 5-g extracts did not exhibit long-lived integrity. NBS SRM 1645 5-g extracts evidenced precipitation on standing after 1 day from filtration. This difficulty was not encountered for the program samples and clearly indicates the need of an additional standard reference material for the analysis of lead in soil. A future soil program would certainly benefit from a standard reflecting more closely the matrix of a nonaquatic soil. It is possible that soil archived from the Dallas Lead Study could provide such a reference.

Data handling has a significant impact on a project of this scope. The transcription error possibilities are escalated by the redundant handling of sample information. In addition, a marked reduction in cost-effectiveness occurs when computerized data handling is reduced to a manual operation. A great deal of effort was expended during the Dallas Lead Study to transcribe data stored on the computer to keypunch analytical data report forms. The importance of continuity from the field to the Agency cannot be overemphasized. A future program would significantly benefit from computer linkage between field and laboratory, laboratory and Agency, and Agency and field. Field sample identification would follow to the laboratory where laboratory tracking numbers would be assigned. Daily transmittals of all pertinent sample information would then proceed to the Agency.

While the quality control requirements of the Dallas Smelter Study were ot unreasonable considering the program objective, the analyses required to onitor the protocol digestion methodology, "total recoverable" and reextraction procedures represent time-consuming operations. Future soil sample programs would benefit from the extraction efficiency data base generated during this study. Preliminary investigation would likely suffice to identify soils in other geographical locations as having similar extraction behavior. It would therefore be possible to decrease the frequency of or eliminate these two analytical categories.

The most critical parameter affecting the efficiency and cost-effectiveness of a program requiring high sample throughput and high level of quality control is the overall organization of discreet project tasks. It is imperative to the successful completion of a project of this genre that the identification of task requirements and the organization of personnel be addressed sufficiently ahead of sample flow to be in place.

The soil analytical segment of the Dallas Lead Study was organized by the assignment of distinct individuals to particular sample handling tasks. This proved to be the most efficient approach as evidenced by escalation of analyst performance over the course of the study. A key position in the organization was the "floating" analyst. This individual was assigned the responsibility of assessing problem areas in the overall pace of the analytical team. Task performance was reviewed on a daily basis. It was this analyst's charge to assist in the task where sample flow was impeded.

The only deviation from the originally provided EPA analytical protocol, Appendix A, was the slurrying of dry sample with 25 ml DI water with subsequent addition of 25 ml conc. HNO_3 acid. This was performed in lieu of the addition of 50 ml of 1:1 HNO_3. The result affected was to reduce the violent frothing and possible loss of sample. The implementation of prior sample wetting significantly improved sample throughput.

APPENDIX J

DALLAS LEAD SMELTERS LAND USE ANALYSIS

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DALLAS LEAD SMELTERS LAND USE ANALYSIS

Preliminary Results

by

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ABSTRACT

A five category land use analysis and area measurement summary was carried out by the Environmental Monitoring Systems Laboratory, Las Vegas, for the Environmental Protection Agency, Region 6. Area measurements were performed on the Level 1, five-category classification system within a 1 square mile study area surrounding two lead smelter sites in Dallas, Texas. The preliminary land use analysis and associated measurements will assist in cost assessments for lead contamination cleanup of both study areas.

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METHODOLOGY

AERIAL PHOTOGRAPHY AND PHOTO INTERPRETATION

Two enlarged color prints (approximate scale 1:9,600 or 1 inch equals 800 feet) covering the D.M.C. and R.S.R. lead smelter sites provided the basis for land use interpretation and area measurements. The photographs were acquired in February 1982 during the vegetation die-back period, which partially accounts for the overall dense (dark) photographic tones evident in the photography. Seasonal effects on the remainder of ground features were minimal.

A mylar overlay was placed over each photo and appropriate land use outlined.

LAND USE CATEGORIES

A Level 1, five-category classification was used in land use analysis:

1 - Vegetation (includes trees, shrubs, pastures, and naturally occurring grasslands)
2 - Water (lakes, ponds, rivers, and other standing water areas)
3 - Bare Ground (includes areas mostly devoid of vegetation cover resulting from man's activities and natural processes)
4 - Paved Surfaces (includes highways, roads, parking lots and aprons, and storage areas)

5 - Buildings/Structures (includes residences, industrial buildings and structures - acreage includes lots and land associated with residential development. Residential sidewalks and streets are excluded from this acreage).

GRIDDED OVERLAY

A grid consisting of sixty-four 1/4-square mile (40-acre) cells was placed over the interpreted photo to provide the base for area measurements. The grid is centered on each smelter site. Tables 1 and 2 summarize the results of measurement on all five land use categories present in each cell (A1, A2, B1, B2, etc.).

MEASUREMENT TECHNIQUE

A Bruning Areagraph Chart (No. 4850) was used for area measurement. The dot grid chart was placed over each 40-acre cell, and the total dots per class counted and recorded. These figures were then converted to acreage measurements (nearest tenth of an acre). This method has a 97 percent degree of precision provided the area measured exceeds 12 square inches.

An additional land use classification category, garden areas, was not included in this preliminary analysis because the small photographic scale (1 inch equals 800 feet) was insufficient to permit accurate location and delineation of these features. Additionally, seasonal timing of the overflight (midwinter) resulted in greatly reduced vegetation growth. The final analysis of the Dallas lead smelter study areas will enable delineation of garden areas because the timing of the overflight will coincide with the growing season, and the use of color infrared film (2443), a chlorophyll-sensitive film will be useful in locating small areas of growing, green vegetation.

TABLE 1. R.S.R. STUDY AREA MEASUREMENTS

	Vegetation	Water	Bare Ground	Paved Surfaces	Buildings/ Structures
A1	21.4	1.4	2.0	3.3	11.6
A2	20.1	1.6	.5	4.7	12.9
A3	5.9	1.3	-	6.3	26.5
A4	1.5	-	-	9.1	29.4
A5	5.1	3.4	-	3.6	27.9
A6	28.6	2.2	-	2.8	6.4
A7	19.3	2.0	-	2.2	16.5
A8	24.7	-	-	8.7	6.6
B1	28.8	-	11.2	-	-
B2	31.3	-	1.9	2.1	4.7
B3	23.2	1.0	3.7	2.5	9.6
B4	15.5	-	-	6.5	18.0
B5	7.8	2.5	-	5.6	24.1
B6	28.6	1.8	-	4.7	4.9
B7	6.0	-	-	5.7	28.3
B8	14.3	-	1.0	5.3	19.4
C1	19.0	-	16.5	3.2	1.3
C2	14.9	-	5.9	10.0	9.2
C3	1.2	-	-	6.3	32.5
C4	1.5	-	-	8.5	30.0
C5	-	-	-	9.1	30.9
C6	27.5	4.0	-	2.6	5.9
C7	15.1	10.0	-	4.4	10.4
C8	19.3	-	-	5.9	14.8
D1	26.7	-	-	9.5	3.8
D2	4.8	-	-	25.7	9.4
D3	-	-	-	4.1	35.9
D4	4.0	-	.4	5.3	30.2
D5	2.2	-	1.8	6.8	29.2
D6	5.6	-	-	9.1	25.3
D7	12.3	9.8	.7	15.4	1.8
D8	6.6	-	5.1	15.0	13.2
E1	14.4	-	-	23.4	2.2
E2	23.8	-	-	10.9	5.3
E3	26.8	-	1.6	7.6	4.0
E4	4.4	-	19.7	9.8	6.1
E5	26.8	-	4.0	4.4	4.8
E6	23.0	-	3.4	5.6	8.0
E7	16.6	-	8.7	3.5	11.2
E8	6.1	-	6.0	12.8	15.1

(continued)

NOTE: All figures are given in acres to the nearest tenth.

TABLE 1 (continued)

	Vegetation	Water	Bare Ground	Paved Surfaces	Buildings/ Structures
F1	9.4	-	30.6	-	-
F2	5.6	-	9.0	7.9	17.5
F3	7.0	-	-	19.8	13.2
F4	23.5	-	7.9	6.1	2.5
F5	20.2	-	4.8	4.9	10.1
F6	16.3	-	11.4	6.3	6.0
F7	22.8	-	3.4	7.3	6.5
F8	16.6	-	4.4	7.0	12.0
G1	11.5	-	25.5	3.0	-
G2	12.8	-	19.1	3.2	4.9
G3	25.9	-	7.6	5.0	1.5
G4	12.3	-	18.5	8.4	.8
G5	9.3	-	24.0	6.7	-
G6	28.7	1.0	1.4	8.6	.3
G7	25.1	1.2	2.2	8.5	3.0
G8	33.8	-	1.7	3.5	1.0
H1	29.8	10.2	-	-	-
H2	36.0	-	4.0	-	-
H3	37.1	-	2.9	-	-
H4	32.6	-	.2	3.4	-
H5	26.3	-	7.6	4.1	3.8
H6	6.1	2.6	23.3	-	2.0
H7	5.8	2.0	-	25.7	8.0
H8	13.3	1.2	-	19.1	6.5
					6.4
TOTALS	1052.5	59.2	303.6	450.5	693.3

TABLE 2. D.M.C. STUDY AREA MEASUREMENTS

	Vegetation	Water	Bare Ground	Paved Surfaces	Buildings/ Structures
A1	34.7	2.3	–	3.0	–
A2	32.6	4.4	3.0	–	–
A3	3.0	17.5	19.5	–	–
A4	34.8	3.0	–	2.2	–
A5	24.8	1.4	11.0	–	2.8
A6	3.7	–	6.6	2.0	27.7
A7	1.3	–	–	20.4	18.2
A8	.4	–	–	9.7	29.9
B1	28.0	–	.4	2.8	8.8
B2	35.4	1.4	.7	.6	1.9
B3	30.1	2.0	5.1	.3	2.5
B4	37.3	–	.3	2.4	–
B5	37.7	2.3	–	–	–
B6	23.4	2.2	12.0	–	2.4
B7	22.1	–	9.0	8.4	.5
B8	15.6	–	12.8	3.4	8.2
C1	14.4	–	–	8.0	17.6
C2	–	–	–	4.9	35.1
C3	4.8	–	7.6	5.9	21.0
C4	27.1	–	5.3	3.2	4.2
C5	37.3	–	.4	1.3	.9
C6	37.1	2.8	–	–	–
C7	36.1	1.9	–	1.9	–
C8	23.8	–	13.5	2.6	–
D1	18.6	–	6.6	4.7	10.1
D2	–	–	–	4.7	35.3
D3	–	–	–	6.3	33.7
D4	5.4	–	6.2	3.7	24.7
D5	24.7	–	3.4	3.2	8.7
D6	25.7	5.4	–	–	8.8
D7	25.5	14.5	–	–	–
D8	31.5	2.0	–	6.5	–
E1	7.2	–	3.4	3.8	25.5
E2	7.0	–	–	5.6	27.3
E3	2.5	–	2.5	4.5	30.5
E4	6.1	–	–	3.2	30.7
E5	25.3	–	5.4	6.6	2.7
E6	6.2	–	–	.6	33.2
E7	31.7	1.5	–	–	6.8
E8	33.7	2.5	–	3.8	–

(continued)

NOTE: All figures are given in acres to the nearest tenth.

TABLE 2 (continued)

	Vegetation	Water	Bare Ground	Paved Surfaces	Buildings/ Structures
F1	2.8	–	–	6.6	30.6
F2	8.8	–	–	6.3	24.9
F3	26.9	–	–	2.8	10.3
F4	26.3	–	.5	9.4	3.8
F5	33.9	2.6	–	3.0	.5
F6	25.8	–	1.0	3.8	9.4
F7	15.6	–	–	1.5	22.9
F8	26.7	9.3	–	4.0	–
G1	–	–	–	5.1	34.9
G2	5.9	–	–	5.9	28.2
G3	32.6	–	.3	1.0	6.1
G4	22.6	3.5	7.3	3.7	2.9
G5	35.5	3.4	–	1.1	–
G6	31.7	4.6	.4	3.3	–
G7	36.1	–	3.9	–	–
G8	28.0	10.3	1.7	–	–
H1	2.0	–	–	7.6	30.4
H2	12.3	–	–	5.7	22.0
H3	33.8	–	–	1.3	4.9
H4	15.4	11.3	–	2.9	10.4
H5	16.1	23.9	–	–	–
H6	30.5	4.7	4.8	–	–
H7	37.4	–	.6	1.5	.5
H8	27.3	–	12.2	.5	–
TOTALS	1330.6	140.7	167.4	217.2	702.4

APPENDIX K

DALLAS LEAD STUDIES: SOIL HOMOGENEITY, METHODS EVALUATION, AND SOIL SAMPLING EQUIPMENT DECONTAMINATION TEST RESULTS

Homogeneity Test

Eleven soil samples, 4 from RSR, 4 from DMC and 3 from the REF study areas were collected. Each sample was dried, sieved, mixed and split into three equal portions as identified in Appendix D, Attachment 1. The results identified below show the lead concentration (μg/g) of each portion, the mean ($\bar{X}$) of the three portions and their Relative Standard Deviation (%).

GCA Number	Site I.D. Code Number	Concentration Lead (μg/g)	$\bar{X}$	Relative Standard Deviation (%)
23132	RSR-E11-X	367		
23133	RSR-E11-Y	349	354	3.1
23134	RSR-E11-Z	347		
23135	RSR-K14-X	102		
23136	RSR-K14-Y	94.9	98.4	3.6
23137	RSR-K14-Z	98.3		
23138	RSR-B04-X	64.0		
23139	RSR-B04-Y	64.3	63.8	1.1
23140	RSR-B04-Z	63.0		
23141	RSR-008-X	139		
23142	RSR-008-Y	141	140	0.71
23143	RSR-008-Z	140		
23144	DMC-D04-X	78.3		
23145	DMC-D04-Y	78.2	78.4	0.34
23146	DMC-D04-Z	78.7		
23147	DMC-N07-X	52.7		
23148	DMC-N07-Y	49.7	50.5	3.8
23149	DMC-N07-Z	49.1		
23150	DMC-L11-Y	273		
23151	DMC-L11-Z	373	344	18
23152	DMC-L11-X	385		
23153	DMC-D14-X	49.7		
23154	DMC-D14-Y	44.1	48.7	8.5
23155	DMC-D14-Z	52.2		
23156	REF-1-X	34.7		
23157	REF-1-Y	36.2	35.7	2.4
23158	REF-1-Z	36.1		
23159	REF-2-X	35.8		
23160	REF-2-Y	36.9	37.7	6.4
23161	REF-2-Z	40.4		
23162	REF-3-X	451		
23163	REF-3-Y	386	417	7.8
23164	REF-3-Z	413		

The following are the results of the reextraction and total recoverable analysis.

1.	Reagent Blank:	< 0.5 μg/ml
2.	Calibration Control Sample (WP 475, Concentrate 3 Diluted 1:10)	Reported: 2.5 μg/ml Expected: 2.5 μg/ml Percent Recovery: 100
3.	Spiked Extract Analysis Sample No. 23135 (RSR-K14-X)	Reported: 5.4 μg/ml Expected: 5.2 μg/ml Percent Recovery: 104
4.	Reextraction of Sample Residue Sample No. 23134 (RSR-E11-Z)	Original Extraction: 347 μg/g Residue Extraction: < 10 μg/g Percent Originally Extracted: 100
5.	Total Recoverable Analysis Sample No. 23134 (RSR-E11-Z)	Original Result: 347 μg/g "Total" Method Result: 374 μg/g Percent Difference: 7.5
6.	Laboratory Control Sample NBS SRM 1645, River Sediment	Reported A: 735 μg/g B: 698 μg/g Expected A: 714 μg/g B: 714 μg/g Percent Recovery A: 103 B: 98
7.	Split Extract Analysis Sample No. 23135 (RSR-K14-X)	Original Extract Result: 102 μg/g Split Extract Result: 106 μg/g Percent Difference: 3.9
8.	Spiked Sample Analysis Sample No. 23135 (RSR-K14-X)	Spiked Sample Result: 947 μg/g Unspiked Sample Result: 74.7 μg/g Spike Added: 808 μg/g Percent Recovery: 108

DALLAS SMELTER STUDY TOTAL RECOVERABLE ANALYSIS REPORT

GCA Sample Number	Site ID Code Number	Original Extraction Concentration Lead (μg/g)	"Total" Method Results Lead (μg/g)	Percent Difference
23135	RSR-K14-X	107	102	4.7
23138	RSR-B04-X	66.4	69.6	3.4
23141	RSR-088-X	139	132	5.2
23144	DMC-D04-X	88.9	85.7	2.2
23147	DMC-N07-X	57.2	52.7	8.2
23151	DMC-L11-Z	596*	376	45
23155	DMC-D14-Z	55.2	53.2	3.7
23156	REF-1-X	40.6	38.7	4.8
23159	REF-2-X	43.2	44.3	2.5
23162	REF-3-X	386	401	3.8

*Reflects inhomogeneity of original sample.

DALLAS SMELTER STUDY REEXTRACTION ANALYSIS REPORT

GCA Sample Number	Site ID Code Number	Original Extraction Concentration Lead (μg/g)	"Total" Method Results Lead (μg/g)	Percent Difference
23135	RSR-K14-X	107	< 10	100
23138	RSR-B04-X	66.4	< 10	100
23141	RSR-088-X	139	< 10	100
23144	DMC-D04-X	88.9	< 10	100
23147	DMC-N07-X	57.2	< 10	100
23151	DMC-L11-Z	596	< 10	100
23155	DMC-D14-Z	55.2	< 10	100
23156	REF-1-X	40.6	< 10	100
23159	REF-2-X	43.2	< 10	100
23162	REF-3-X	386	< 10	100

Dallas Lead Smelter Study
Soil Sample Quality Assurance

1.	Reagent Blank:	< 0.5 μg/ml
2.	Calibration Control Sample (WP 475, Concentrate 3 Diluted 1:10)	Reported: 2.6 μg/g Expected: 2.5 μg/g Percent Recovery: 104
3.	Calibration Control Sample (WP 475, Concentrate 3 Diluted 2:10)	Reported: 5.2 μg/ml Expected: 5.0 μg/ml Percent Recovery: 104
4.	Laboratory Control Sample NBS SRM 1645 River Sediment	Reported: 785 μg/g Expected: 714 μg/g Percent Recovery: 110
5.	Spiked Extract Analysis Sample No. 23144 (DMC-D04-X)	Reported: 4.6 μg/g Expected: 4.6 μg/g Percent Recovery: 100
6.	Spiked Total Recoverable Extract Analysis Sample No. 23138 (RSR-B04-X)	Reported: 3.1 μg/g Expected: 3.1 μg/g Percent Recovery: 100
7.	Split Extract Analysis Sample No. 23141 (RSR-008-X)	Original Extract Result: 131 μg/g Split Extract Result: 132 μg/g Percent Difference: < 1

Decontamination Test

The following are concentrations of lead found in 100 ml extracts of water. Number 1 through 11 are deionized distilled water (DDI) rinses of the soil corers used to collect the soils for the homogeneity test. Numbers 12, 13, and 14 are samples of DDI water that was not used in the rinsing process.

Sample ID No.	Extract Concentration (μg/ml)	Lead In Sample (μg)	Possible Lead Contamination Assuming a 150 gram Sample (μg Pb/gm)
1. DMC-N07B	.21	21	0.14
2. DMC-D04B	.18	18	0.12
3. DMC-D14B	.10	10	0.07
4. DMC-L11B	.11	11	0.07
5. RSR-008B	.18	18	0.12
6. RSR-B04B	.24	24	0.16
7. RSR-E11B	.18	18	0.12
8. RSR-K14B	.23	23	0.15
9. REF-18	.18	18	0.12
10. REF-2B	.19	19	0.13
11. REF-3B	.20	20	0.13
12. DMC-BLANK	.12	12	0.08
13. RSR-BLANK	.16	16	0.11
14. REF-BLANK	.15	15	0.10

APPENDIX L

SAMPLE BANK AUDIT REPORT

PROGRAM AUDITS

INTRODUCTION

A system audit is an overall evaluation of a project to:

- check on the use of appropriate quality control (QC) measures
- identify any existing quality problems
- initiate corrective action if a problem is identified
- follow-up on any corrective action previously implemented

The purpose of the audit is to ensure that the quality system planned for the project is in place and functioning well.

The auditor reviews the Work Plan Summary, Test Plan and QA Project Plan, and Monthly Reports and then discusses the current status of the project and any problems encountered with the GCA Project Manager and Principal Investigators. Sample chain-of-custody procedures and raw data are checked as appropriate and results on the blind QC samples routinely inserted in the sample load by the Laboratory QC Coordinator, not recognizable to the analyst as QC samples, are reviewed. Spot-checks of sampling and analysis calculations and data transcription are performed.

Audit results are promptly summarized in memo format and distributed to the GCA/Technology Division General Manager and Contracts Administrator as well as the appropriate Department Managers and program and project staff. Each person working on the project receives a copy. This summary audit report is included in the Draft Final Report distributed to EPA.

PROGRAM-SPECIFIC AUDIT PROCEDURES

Two program audits of the Analysis of Dallas Soil Samples Work Assignment were conducted. The Program Manager conducted an onsite audit of Dallas Sample Bank operations, and the QA Manager performed an in-house audit covering both field and analytical work. Both audits focused on the implementation of EPA's EMSL-LV QA Project Plan for the Dallas Lead Study DA 68-02-3168 which incorporates GCA's QA Project Plan as an appendix. The remainder of this section describes audit procedures used and results found in these program audits. Appendix C contains the audit work sheets.

In addition, this section describes actions taken by GCA's Dallas Sample Bank staff to improve documentation and verify the exact number of soil samples processed. These activities took place in-house after the onsite Dallas Sample Bank operation was completed. They are presented under the heading Dallas Sample Bank Logbook Verification.

Descriptions of the Program Audits and Sample Bank Logbook Verification are presented in chronological order as follows:

- Onsite Sample Bank Audit
- Dallas Sample Bank Logbook Verification
- In-house Dallas Lead Monitoring Program Audit

ONSITE AUDIT

An audit of the onsite Sample Bank was conducted by the GCA Program Manager during the period September 6 and 7, 1982. The primary objective of this audit was to determine the status of all Sample Bank documentation and archived samples. Emphasis was placed on:

- Verifying that the documentation was in order and sufficient to establish the disposition of any sample collected
- Determining any discrepancies that currently existed and initiate corrective action as appropriate
- Verifying that the recordation of QC/QA measures (blanks, spikes, blinds) was in accordance with the QA Plan
- Establishing the final disposition and mechanics of transfer of all Sample Bank holdings upon termination of the operation.

The first step of the audit was to inventory the Sample Bank records and archived samples. The records inspected were:

- Chain-of-custody forms
    - Field forms
    - Analysis forms
- Sample tags
    - Field tags (white, brown, green)
    - Analysis tags (white, green)

- Analysis forms
  - Individual samples (GCA and ESA)
  - Batch sheets
- Shipment forms
- Logbooks
  - Blood (1)
  - Grids and vegetation (1)
  - Residential soils and dusts (3)
  - Daily log (1)

This initial inspection revealed that all of the required documentation had been maintained in an orderly fashion. Each of the recordation items was properly categorized, and cross-checking could be easily performed. In addition, with the exception of the field sampling tags, the recordation conformed to strict document control protocols. Because the sample tags were not preserialized, the disposition of all existing tags (not samples) could not be determined. This fact is of little significance vis-a-vis sample chain of custody or data quality.

The archived samples inspected can be categorized as follows:

- Vegetation archives
- Soil archives
- Excess dust blanks
- Excess soil blanks

An audit of 100 percent of the vegetation archives was conducted. It was verified that the appropriate vegetation sample existed for each entry in the vegetation logbook. Also as part of this process, the field sample tags were replaced by the appropriate analytical tags, and chain-of-custody forms were prepared in order to transfer the samples. Prior to termination of the Sample Bank activities, the transfer was executed with City of Dallas personnel.

An audit was then conducted on the soil sample archives. This audit entailed a comparison of the logbook entries against the archived samples. All samples were first grouped in 100 (maximum) sample sequences. All split samples and blanks were identified for each sequence examined. The totals for splits and blanks were used to determine the number of samples that should exist for each sequence. If the expected and actual values agreed, no further examination was made. If a discrepancy was found, a more detailed examination was made. This procedure allowed for the determination of the status of the soil archives and a detailed examination of the logbook recordation for the soil samples.

This procedure was performed on 23 of the total of 33 sample sequences during the audit. (It was performed on the remaining 10 sequences prior to the termination of the Sample Bank activities.) The completed work sheets for this procedure are included in Appendix C. A summary of QA/QC data tallied for the first 23 sequences is given in Table 6-1. Because three logbook entries are generated for each split sample collected, the 330 entries represent 100 field samples. This results in 110 splits and 146 blanks for 1900 field samples. These totals agree well (a slight excess) with the planned QA/QC.

It should be noted that Sequence Numbers S00330-S00339 do not exist in the logbook; i.e., Sample Number S00340 follows the entry for S00329. In addition, the following discrepancies are noted:

- S00164 was DMC-G03, a designated split. The sample was not split three ways as intended; it was split in two. One portion was shipped to NEIC on Chain of Custody No. 8102 and Analytical Tag No. 08610. The other portion was archived and is in the possession of the Sample Bank.
- S00173 originally appeared on two Analytical Chain of Custody forms, No. 8100 and No. 8106. This duplication was caught after shipment when the shipment containing chain of custody sheet entry was removed. While performing the archive inventory, three samples labeled S00173 were found. Two were labeled with archive tags, one was labeled on Analytical Tag No. 08949. Apparently a duplication was involved in this mix-up, possibly one of the samples was one of the soil samples lost. It is not possible to verify this, however.
- S00291 was found to be duplicated in the soil archive. The duplicate archive sample is in the possession of the Sample Bank. An archive portion was missing for S00191. It is quite possible one of the two archive portions should have been labeled as S00191.
- S11131 was found to be duplicated in the archive. The archive tag was originally completed as S1131, obviously an error. It was changed to S11131 and initialed. It is more likely that this aliquot is S11331 which is missing an aliquot in the archive. This aliquot is currently in the possession of the Sample Bank.
- S11175 also appeared in duplicate in the archive. One of the two aliquots is in the possession of the Sample Bank. The reason for this duplication is not known.
- S11332 also appeared in the archive in duplicate. One aliquot is in the possession of the Sample Bank. The origin of this duplication is unclear.

TABLE 6-1. SUMMARY OF QA/QC SAMPLES AUDITED

Sequence No.	Split samples	Blanks	Sequence totals
S000	5	1	100
S001	6	0	100
S002	12	0	100
S003	27	25	90[a]
S004	46	9	100
S005	9	0	76[b]
S105	4	0	100
S106	5	0	100
S107	5	0	100
S108	4	2	100
S109	17	3	100
S110	24	5	100
S111	24	3	100
S112	16	13	100
S113	15	10	100
S114	15	12	100
S115	12	6	100
S116	15	8	100
S117	12	13	100
S118	15	9	100
S119	12	10	100
S120	15	8	100
S121	15	9	100
Totals	330	146	2266

[a] Numbers S00330-S00339 do not exist (see text).

[b] End of series.

- S12564 is another duplicate from the archive, the origin of which is unknown, with one of the aliquots in the possession of the Sample Bank.

- The nine samples listed below are missing an aliquot in the archive. Possibly they were mislabeled or sent to the analytical laboratory without an aliquot archived.

  - S00157
  - S00191
  - S11229
  - S11330
  - S11331
  - S11333
  - S11587
  - S11676
  - S11987

In addition to the inventory of the soil sample archive, excess field blanks and all vegetation samples were transferred to the archive and appropriate transfer documents prepared. The inventory process verified that the "excess" samples were actually blanks in excess of the planned QA/QC.

DALLAS SAMPLE BANK LOGBOOK VERIFICATION

Subsequent to the completion of field operations, in-house quality assurance efforts at GCA/Technology Division facilities in Bedford, MA, were performed under the direction of the GCA Field Sample Bank Manager. This activity was twofold. From September 20-22, 1982, incomplete entries in the master logbooks, remaining from the initial period of field Sample Bank operation prior to GCA management, were completed and verified against the Sample Bank document file.

The one exception to this procedure concerned the blood sample master logbook. All blood sample vials had affixed to them an adhesive label with the site and sample identification code typed on it. This label remained on the vial from the time the sample was collected to its receipt by the analytical laboratory. Therefore, for this sample category, identification of the sample origin remained on the sample through all field Sample Bank handling. In light of this unique procedure, the entry of blood sample data in the master logbook prior to GCA management was abbreviated.

Specifically, for the first 210 blood samples entered in the logbook, the field Chain of Custody number, the ESA Laboratory Analysis Data Form number, and the shipping date were frequently omitted. Also, for these first 210 samples, no Analytical Chain of Custody forms were completed. Due to the direct labeling of all blood samples with the site and sample identification code, the absence of any inquiries from the ESA laboratory, EPA EMSL-LV, or RTI regarding these samples, and the estimate of the time required to complete these entries, master logbook entries remain as described. The omitted data remain on the appropriate documents in the Sample Bank files. If at a later date it is deemed necessary to complete these logbook entries, it can be accomplished.

The second activity performed involved the tabulation of the number of samples processed by the field Sample Bank. Under the direct supervision of the GCA field Sample Bank Manager, a detailed listing of all samples was compiled by hand tallying the samples by categories. This was accomplished by proceeding page by page through all master logbooks. The results were compiled and included in Section 1 of the Final Report.

The computed total of soil samples sent to GCA as listed in preliminary compilation did not agree with an independently derived total of samples received by the GCA Analytical Laboratory. To resolve this discrepancy, this listing was completely recounted. The procedure for this recount involved first checking off by hand each Sample Bank Number entered on each Analytical Chain of Custody form submitted to the GCA Laboratory. A tabulation of the total number of samples per shipment in the following four categories was prepared: grid soil samples, residential soil samples, field blanks, and Sample Bank blanks.

This second listing was found to agree exactly with the total number of samples received by the GCA Analytical Laboratory. In addition, an accounting of all other categories of soil samples logged in the soil master logbooks was prepared. This allowed the GCA field Sample Bank Manager to balance the total number of logbook entries against this list.

IN-HOUSE DALLAS LEAD MONITORING PROGRAM AUDIT

A system audit of work on the project was conducted by the QA Manager at GCA's Bedford, MA, facility on 30 September and 5-6 October 1982. Although the Dallas Sample Bank operation was completed and had been audited onsite, it was included in this qualitative review of GCA's work on the Dallas Lead Monitoring Program. The remainder of this section describes audit procedures and results and includes the summary memo audit report.

Dallas Sample Bank Operations

GCA's work at the field Sample Bank was audited through discussions with the Sample Bank Manager and review of the Field Log, the Sample Bank Logbooks for different media, and the custody forms.

The Dallas Sample Bank Manager indicated that problems in chain-of-custody procedures were recognized when GCA took over operation of the Dallas Sample Bank. The problems were attributable to an initial staff inexperienced in custody requirements, and to changes in operating procedures initiated by EPA-Las Vegas and Research Triangle Institute personnel. The problems identified and the actions taken to correct them were documented in the Field Log in an appropriate manner.

Field Log--

This log was maintained by the GCA person in charge at the Dallas Sample Bank; two staff members handled this responsibility during the 8-week period of GCA management. A new dated page was started for every working day listing GCA staff working and the hours worked. Clear, concise entries detailed events of the day (such as numbers of samples processed), problems encountered, and actions taken to solve them. This log provides excellent documentation of GCA's operation of the Sample Bank.

Sample Bank Logs--

The audit review of these logs showed complete sample information entered. Most changes were properly made by crossing out so the original entry was still visible, and initialing the correct information. A few improper corrections using typewriter correction fluid which obliterated the original entry were not initialed; the field Sample Bank Manager stated these were made by non-GCA personnel without his knowledge or approval.

The Blood Log was checked to verify the early use of ESA Laboratory forms instead of custody forms to record shipment of samples and GCA's utilization of preserialized chain of custody forms for blood samples. Appendix C shows an ESA Laboratory form corresponding to Master Log pages 8 and 9 and the program Chain of Custody form corresponding to Logbook pages 32 and 33.

The Grid Soils Log was checked for identification of split and duplicate samples, and field and Sample Bank blanks; this was consistently done from 20 July 1982. The Residential Soils Log was not as consistent in designating these types of samples until 3 August 1982.

Sample Bank Custody and QC Requirements--

Auditing of the maintenance of sample chain of custody involved checking the use of these logs and GCA's internal laboratory custody procedures and forms and is discussed under Laboratory Analysis Operations. The same is true of the requirements for duplicate and blank samples to be generated and/or processed by the field Sample Bank. As noted above, the field Sample Bank Logs documented the existence of these QC samples; Appendix C to this report provides an example of the check of duplicate field samples from the Dallas Sample Bank Log to the final Analysis Data Reporting Form.

GCA Laboratory Analysis Operations

GCA's analytical work was audited by discussions with the Laboratory Analysis Principal Investigator, the GCA Sample Bank Manager, and individual analysts, and by review of Custody Forms, GCA Soil Sample Travelers, Analysis Data Reporting Forms, and Analytical Data Transmittal Packages.

The Principal Investigator described the specialized assignments for the dedicated analytical team and the streamlined data handling which facilitated the high throughput of samples. All analytical raw data was made available, and review of these data is discussed under the individual topics listed below. She affirmed good interaction between EPA and GCA analytical staff and between GCA's Dallas Sample Bank staff and the analytical staff.

Sample Homogeneity/Extraction Efficiency--

The homogeneity study was conducted in July on eleven samples, ranging in concentration from approximately 30-400 micrograms of lead per gram of sample. Triplicate aliquots were carried through the entire analytical protocol; the precision of analytical results was excellent. Seven samples had a relative standard deviation (RSD) of less than 4 percent, three ranged from 6 to 8.5 percent RSD, and one sample had an 18 percent RSD.

Extraction efficiency data on the protocol extraction of the residue from the original extraction of ten samples showed less than 10 micrograms lead per gram of original sample. Total recoverables data comparing a nitric acid plus peroxide digestion with the protocol nitric acid procedure demonstrated very close agreement between analytical results on aliquots of the same sample digested by both methods. Raw data for ten samples showed eight had less than 5 percent difference between the two aliquots, one sample had an 8 percent difference, and one, apparently not a homogeneous sample, had a 45 percent difference.

The results of these studies attested to the conscientious work of the field Sample Bank staff and the Bedford analytical staff working on sample preparation and analysis. Also, based on these studies, the reextraction step was eliminated in late August and, in September, the frequency of Total Recoverable digestions was decreased.

Sample Chain of Custody--

Laboratory custody procedures were audited by spot-checks of the Dallas Lead Chain of Custody Records, GCA's Sample Bank Master Log and Custody Record Books, GCA Soil Sample Travelers, and Dallas Lead Monitoring Project Analysis Data Reporting Forms. It was easy to follow individual samples from receipt through laboratory work to final reporting. No transcription errors were found in spot-checks of the assigned GCA Control Numbers for proper cross-referencing with the Dallas Sample Bank sample numbers. The Sample Travelers were signed and dated by the analysts to document custody transfers within the laboratory.

The overall chain of custody from sample collection, identification and transfer of sample custody to the Dallas Sample Bank through GCA's final reporting of results to EPA was audited when checking on sampling and analytical QC requirements.

Sampling and Analytical QC Requirements--

Analytical Data Packages which included a cover letter, the EPA Analysis Data Reporting Forms, and the associated Soil Sample Quality Assurance Forms were reviewed to check on the implementation of EPA's QC requirements. The Dallas Sample Bank Soil Logs were used to identify duplicate and split field samples, field and Sample Bank blanks, and follow these samples through the system to final reporting of results. It was easy for the auditor to do this and apparent that the analysts could not have done this. The GCA Soil Sample Travelers identified samples only by GCA Control Number; the analysts did not have access to the Dallas or GCA Sample Bank Logs to relate the GCA Control Numbers to Dallas Sample Bank Numbers. In all cases checked, analytical results were reasonable for the type of sample.

Appendix C to this report provides an example of this audit procedure for the block of samples reported in QC Report No. 8. The Analysis Data Reporting Forms show the auditor's designation of sampling sites using the Dallas Sample Bank Logs to identify splits from one field sample. Sample Bank Numbers S00415 (GCA 23452) and S00416 (GCA 23453) were so identified; the lead analysis results were 93.0 and 95.0 micrograms/gram, respectively.

The Soil Sample Quality Assurance Form, QC Report No. 8, was checked against the raw data on the appropriate Sample Traveler. The AA calibration data were written in on the back of the Traveler so that the curve could be constructed and sample concentrations calculated. An internal laboratory correction was properly made on Sample S00089.

Sample S00110 (GCA 23457) wrs designated by the analytical sequence to be utilized as the Spiked Sample and Total Recoverable. All original analyses performed on this particular sample showed evidence of gross sample imhomogeneity as indicated by extreme divergence in the data. Corrective action dictated by the QA plan for data outside the acceptance limit in these two categories was to analyze a second sample. Samples were arbitrarily chosen to fulfill the QA requirements of the data set. Sample S00078 (GCA 23467) was utilized for Total Recoverable analysis and S00089 (GCA 23456) for Spiked Sample analysis. In addition a second aliquot of S00110 was carried through the analytical protocol. These analyses were performed August 26, 1982 and are recorded in the laboratory page shown in Appendix C.

No uncorrected calculation or transcription errors were found in this spot-check of Analytical Data packages.

The summary report of this audit was distributed to the listed senior GCA technical and management staff and all members of the project team by the QA Manager. Through an oversight, this more detailed Audit Report incorporating the summary memo was not prepared for the Draft Final Report distributed to EPA. It is presented here.

GCA Technology Division

213 Burlington Road
Bedford, Mass. 01730
(617) 275-5444

To: 1-619-080 File

Date: 7 October 1982

From: R.M. Ellersick

Subject: QA Audit - Analysis of Dallas Soil Samples

An audit was conducted on 30 September and 6 October 1982 to check on the use of appropriate QC measures on this project. The data reviewed included:

- the project Field Log maintained at the Sample Bank
- several Sample Bank Logs
- Analysis Data Reporting Forms
- GCA Soil Sample Travelers
- Analytical Data Transmittal Packages
- Monthly progress reports

The Field Log provided excellent documentation of activity at the Dallas Sample Bank. It was maintained by the onsite GCA Sample Bank Manager and a new, dated page was started on each working day. Clear, concise entries detailed events of the day, samples shipped, problems encountered, etc.

The Sample Bank Logs followed the EPA heading format with minor differences for each medium sampled; sample information was completely entered. The July monthly report detailed modifications to the initial operating procedures instituted by the GCA staff to improve chain-of-custody recordkeeping and document accountability. Some of these modifications necessitated changes to log book entries which were made by crossing out the original entry, entering the correction and initialing it. The log books contain a few improper corrections - original entries obliterated with typewriter correction fluid. GCA's Dallas Sample Bank Manager states these were made by non-GCA personnel without his knowledge or approval.

Spot checks of the analytical data sheets listed above showed them to be complete and consistent with each other and with the Sample Bank Log. Internal laboratory custody transfers were documented and data entry was handled in an efficient manner. The required QC procedures were followed and clearly documented. Specific samples could be readily traced by the auditor from receipt at the Sample Bank through reporting of results to EPA. This was done for several samples and no unreasonable or missing data was uncovered.

The results of the soil sample homogeneity tests and the extraction efficiency evaluation were reviewed. The excellent precision achieved on the homogeneity tests demonstrates the conscientious work done by both the Sample Bank and the laboratory staff in handling the samples. The extraction efficiency results again affirm careful analytical work.

The project staff is to be commended for their work, especially impressive in view of the high volume of samples handled.

RoseMary Ellersick
RoseMary Ellersick
QA Manager

RME/cf

cc: A. Engelman
T.G. Hopper
K.T. McGregor
L.M. Seale

APPENDIX C

AUDIT WORKSHEETS

Appendix C-1

Work Sheets for Archive Soil Sample Inventory

Legend

X - Blank

$\underline{\overline{X}}$ - Split Sample

Appendix C-2

Inhouse Program Audit

- ESA Laboratory Analysis Data Form
- Dallas Blood Log (pages 8 and 9)
- Dallas Lead Chain of Custody Form
- Dallas Blood Log (pages 32 and 33)
- Analysis Data Reporting Forms for QC Report No. 8
- Soil Sample Traveler for QC Report No. 8
- Laboratory Notebook Page for QC Report No. 8 Corrective Action
- Soil Sample Quality Assurance QC Report No. 8

APPENDIX C-1

WORK SHEETS FOR ARCHIVE SOIL SAMPLE INVENTORY

ARCHIVE SOIL SAMPLE INVENTORY SHEET 7/1/82

BOX SEQUENCE NOS. S00000 – S00099

NUMBER IN BOX 94 (BY COUNT) 94 (BY LOG BOOK)

								SUMMARY
	S00000		25		50		75	S00000 – S00005
	01		26		51		76	S00007 – S00011
	02		27		52		77	S00013 – S00027
	03	☒	28		53		78	S00029 – S00040
	04		29		54		79	S00042 – S00047
	05		30		55		80	S00049 – S00056
☒	06		31		56		81	S00058 – S00099
	07		32	☒	57		82	
	08		33		58		83	
	09		34		59		84	
	10		35		60		85	
	11		36		61		86	
X	12		37		62		87	
	13		38		63		88	
	14		39		64		89	
	15		40		65		90	
	16	☒	41		66		91	
	17		42		67		92	
	18		43		68		93	
	19		44		69		94	
	20		45		70		95	
	21		46		71		96	
	22		47		72		97	
	23	☒	48		73		98	
	24		49		74		S00099	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82

BOX SEQUENCE NOS. S00100 - S00199

NUMBER IN BOX 92 (BY COUNT) 94 (BY LOG BOOK)

									SUMMARY
S00100		- 25			- 50			- 75	
- 01		- 26			- 51			- 76	
- 02		- 27			- 52			- 77	
- 03		- 28			- 53			- 78	
- 04		- 29			- 54			- 79	
- 05	☒	30			- 55			- 80	
- 06		- 31			- 56			- 81	
- 07		- 32	MISSING	X	57			- 82	
- 08	☒	33			- 58			- 83	
- 09		- 34		☒	59			- 84	
- 10		- 35			- 60			- 85	
- 11		- 36			- 61			- 86	
- 12		- 37			- 62			- 87	
- 13		- 38			- 63			- 88	
- 14		- 39	NOT SHIPPED [illegible]	☒	■ 64			- 89	
- 15		- 40			- 65			- 90	
- 16		- 41			- 66	MISSING	X	91	
- 17	☒	42			- 67			- 92	
- 18		- 43		☒	68			- 93	
- 19		- 44			- 69			- 94	
- 20		- 45			- 70			- 95	
- 21		- 46			- 71			- 96	
- 22		- 47			- 72			- 97	
- 23		- 48			- 73			- 98	
- 24		- 49			- 74			S00199	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82

BOX SEQUENCE NOS. S00200 - S00299

NUMBER IN BOX 88 (BY COUNT) 88 (BY LOG BOOK)

									SUMMARY
	S00200		25		50			75	
	01		26	X	51			76	
	02		27		52			77	
	03		28		53			78	
	04		29		54		X	79	
	05		30		55			80	
	06		31		56			81	
	07		32		57			82	
	08		33		58			83	
	09		34		59		X	84	
	10		35		60			85	
	11		36		61			86	
	12		37		62			87	
	13		38		63			88	
	14		39		64			89	
	15		40		65			90	
	16		41		66	pur'd		91	
X	17		42	X	67			92	
	18		43		68		X	93	
	19		44		69		X	94	
	20		45		70		X	95	
	21		46		71	PRELIM STUDY	X	96	
	22		47		72		X	97	
	23		48		73		X	98	
	24		49		74		X	S00299	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82

BOX SEQUENCE NOS. S00300-S00399

NUMBER IN BOX 37 (BY COUNT) 37 KJMcK (BY LOG BOOK)

								SUMMARY
X	S00300		25		50	X	75	
X	01		26		51	X	76	
X	02		27	X	52	X	77	
X	03		28	X	53	X	78	
	04		29	X	54	X	79	
	05	X	30	X	55	X	80	
	06	X	31	X	56	X	81	
	07	X	32	X	57	X	82	
	08	X	33	X	58	X	83	
	09	X	34	X	59	X	84	
	10	X	35	X	60	X	85	
	11	X	36	X	61	X	86	
	12	X	37	X	62	X	87	
	13	X	38	X	63	X	88	
	14	X	39	X	64	X	89	
	15	X	40	X	65	X	90	
	16		41	X	66	X	91	
	17		42	X	67	X	92	
	18		43	X	68	X	93	
	19		44	X	69	X	94	
	20		45	X	70	X	95	
	21		46	X	71	X	96	
	22		47	X	72	X	97	
	23		48	X	73	X	98	
	24		49	X	74	X	S00399	

PRELIM STUDY

NOS. DO NOT EXIST

BLANKS

KJMcK

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82

BOX SEQUENCE NOS. S00400 – S00499

NUMBER IN BOX 43 (BY COUNT) 43 (BY LOG BOOK)

								SUMMARY
	S00400	X	25	X	50	X	75	
	01	X	26	X	51		76	
	02	X	27	X	52		77	
	03		28		53		78	
	04	X	29		54		79	
	05	X	30	X	55		80	
	06	X	31		56	X	81	
	07	X	32		57		82	
	08	X	33		58		83	
	09	X	34		59		84	
	10	X	35		60	X	85	
X	11	X	36		61	X	86	
X	12	X	37		62	X	87	
X	13	X	38		63		88	
X	14	X	39		64		89	
X	15	X	40		65	X	90	
X	16	X	41		66	X	91	
X	17	X	42	X	67	X	92	
Y	18	X	43	X	68		93	
X	19	X	44	X	69		94	
X	20	X	45		70		95	
X	21	X	46		71		96	
X	22	X	47		72		97	
X	23	X	48	X	73		98	
X	24	X	49	X	74	X	S00499	

BLANKS

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/1/82

BOX SEQUENCE NOS. S00500 - S00599 KMcL

NUMBER IN BOX 67 (BY COUNT) 67 (BY LOG BOOK)

								SUMMARY
S00500		25		50		X	75	
01		26		51	END		76	
02		27		52			77	
03		28	X	53			78	
04		29	X	54			79	
05		30	X	55			80	
06		31		56			81	
07		32		57			82	
08		33		58			83	
09		34		59			84	
10		35		60			85	
11		36		61			86	
12		37		62			87	
13		38		63			88	
14		39		64			89	
15		40		65			90	
16		41		66			91	
17		42	X	67			92	
18		43	X	68	NONEXISTENT		93	
19		44	X	69			94	
20		45		70			95	
21		46		71			96	
22		47		72			97	
23		48	X	73			98	
24		49	X	74			S00599	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82

Kimey

BOX SEQUENCE NOS. S10500 – S10599

NUMBER IN BOX 96 (BY COUNT) 96 (BY LOG BOOK)

								SUMMARY
	S10500		25		50		75	
	01		26		51		76	S10500 – S10519
	02		27		52		77	
	03		28		53		78	
	04		29		54		79	
	05		30		55	☒	80	
	06		31		56		81	
	07		32		57		82	
	08		33		58		83	
	09		34		59		84	
	10		35	☒	60		85	
	11		36		61		86	
	12		37		62		87	
	13		38		63		88	
	14		39		64		89	
	15	☒	40		65		90	
	16		41		66		91	
	17		42		67		92	
	18		43		68		93	
	19		44		69		94	
☒	20		45		70		95	
	21		46		71		96	
	22		47		72		97	
	23		48		73		98	
	24		49		74		S10599	

Kimey

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82

BOX SEQUENCE NOS. S10600 – S10699

NUMBER IN BOX 95 (BY COUNT) 95 1ma (BY LOG BOOK)

								SUMMARY
☒	S10600		25		50		75	
	01		26		51		76	
	02		27		52		77	
	03		28		53		78	
	04		29		54		79	
	05		30		55	☒	80	
	06		31		56		81	
	07		32		57		82	
	08		33		58		83	
	09		34		59		84	
	10		35	☒	60		85	
	11		36		61		86	
	12		37		62		87	
	13		38		63		88	
	14		39		64		89	
	15	☒	40		65		90	
	16		41		66		91	
	17		42		67		92	
	18		43		68		93	
	19		44		69		94	
☒	20		45		70		95	
	21		46		71		96	
	22		47		72		97	
	23		48		73		98	
	24		49		74		S10699	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82

BOX SEQUENCE NOS. S10700 - S10799 Kimes

NUMBER IN BOX 25 (BY COUNT) 95 (BY LOG BOOK)

								SUMMARY
☒	S10700		25		50		75	
	01		26		51		76	
	02		27		52		77	
	03		28		53		78	
	04		29		54		79	
	05		30		55	☒	80	
	06		31		56		81	
	07		32		57		82	
	08		33		58		83	
	09		34		59		84	
	10		35	☒	60		85	
	11		36		61		86	
	12		37		62		87	
	13		38		63		88	
	14		39		64		89	
	15	☒	40		65		90	
	16		41		66		91	
	17		42		67		92	
	18		43		68		93	
	19		44		69		94	
☒	20		45		70		95	
	21		46		71		96	
	22		47		72		97	
	23		48		73		98	
	24		49		74		S10799	

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BOX SEQUENCE NOS. S10800 - S10899

NUMBER IN BOX 91 (BY COUNT) ~~93~~ 91 (BY LOG BOOK)

									SUMMARY
☒	S10800		25			50		75	
	01		26			51		76	
	02		27			52		77	
	03		28			53		78	
	04		29	BLANK LOST	X	54		79	
	05		30			55	☒	80	
	06		31	BLANK LOST	X	56		81	
	07		32			57		82	
	08		33			58		83	
	09		34			59		84	
	10		35			60		85	
	11		36			61	X	86	
	12		37			62		87	
	13		38			63		88	
	14		39			64		89	
	15	☒	40			65		90	
	16		41			66		91	
	17		42			67		92	
	18		43			68		93	
	19		44			69		94	
☒	20		45			70		95	
	21		46		X	71		96	
	22		47			72		97	
	23		48	LOST	X	73		98	
	24		49			74		S10899	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82

BOX SEQUENCE NOS. S10900 – S10999

NUMBER IN BOX 80 (BY COUNT) 80 (BY LOG BOOK)

								SUMMARY
X	S10900		25		50		75	
	01		26		51		76	
X	02		27		52		77	
X	03		28		53		78	
X	04		29		54		79	
X	05		30		55		80	
	06		31		56		81	
	07		32		57		82	
	08		33		58	X	83	
	09	X	34		59	X	84	
	10		35		60	X	85	
	11		36		61		86	
	12		37	X	62		87	
	13		38	X	63		88	
	14		39	X	64		89	
X	15	X	40		65		90	
	16	X	41		66		91	
	17	X	42		67		92	
	18		43		68		93	
	19		44		69		94	
X	20		45		70		95	
X	21		46		71		96	
X	22		47		72		97	
X	23		48		73		98	
	24		49		74		S10999	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82
Kimes

BOX SEQUENCE NOS. S11000 – S11099

NUMBER IN BOX 71 (BY COUNT) 71 (BY LOG BOOK)

								SUMMARY
X	S110 00	X	25		50		75	
	01	X	26	X	51		76	
	02	X	27		52		77	
	03	X	28		53		78	
	04	X	29		54		79	
	05	X	30		55		80	
	06	X	31		56		81	
X	07	X	32		57		82	
X	08	X	33		58	X	83	
X	09	X	34		59		84	
	10	X	35		60		85	
	11	X	36	X	61		86	
	12	X	37		62	X	87	
	13	X	38		63		88	
	14		39	X	64		89	
	15		40		65		90	
	16		41		66		91	
	17		42		67		92	
	18		43		68		93	
X	19		44		69		94	
X	20		45		70		95	
X	21		46		71		96	
X	22		47		72		97	
X	23		48		73		98	
X	24		49		74		S11099	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82

BOX SEQUENCE NOS. S1110C – S11199

NUMBER IN BOX 72 (BY COUNT) 73 (BY LOG BOOK)

										SUMMARY
	S11100		X	25		50	Dup		• 75	
	01			26		51		X	76	
	• 02			• 27		52		X	77	
	03			28		53		X	78	
	04		X	29		54			• 79	
	05			• 30	X	55			• 80	
	06	Dupl		• 31	X	56			81	
X	07			• 32	X	57			• 82	
	• 08	Lost	X	33		58			• 83	
	09		X	34		• 59			84	
	10		X	35		• 60			85	
	11		X	36	X	61			86	
	12			• 37		62			87	
	13			38		.63			88	
X	14			39		64			89	
X	15			40		• 65			90	
X	16			41		66			·91	
X	17			42		67			• 92	
X	18			43		68			93	
X	19			44		69			94	
X	20			• 45		70			95	
X	21			46		71			96	
X	22			47		72		X	97	
X	23			48		73		X	98	
X	24			49		74		X	S11199	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET

7/7/82 [illegible]

BOX SEQUENCE NOS. S11200 – S11299

NUMBER IN BOX 70 (BY COUNT) 71 ~~[illegible]~~ [illegible] (BY LOG BOOK)

									SUMMARY
	S112·00			• 25		• 50		• 75	
	• 01		X	26		• 51	X	76	
	• 02			• 27		• 52		• 77	
X	03			• 28		• 53		• 78	
	• 04	missing	X	29		• 54	X	79	
	• 05			• 30		• 55		• 80	
	• 06			• 31		• 56		• 81	
	• 07			• 32		• 57		• 82	
	• 08		X	33		• 58	X	83	
	• 09			• 34	X	59	X	84	
	• 10			• 35		• 60	X	85	
	• 11		X	36		• 61		• 86	
	• 12			• 37	X	62		• 87	
	• 13			• 38	X	63		• 88	
	• 14			• 39	X	64		• 89	
	• 15		X	40		• 65		• 90	
X	16		X	41		• 66	X	91	
X	17		X	42		• 67	X	92	
X	18			• 43		• 68	X	93	
X	19			• 44		• 69		• 94	
X	20			• 45		• 70		• 95	
	• 21			• 46		• 71		• 96	
X	22			• 47		• 72	X	97	
	• 23		X	48	X	73		• 98	
	• 24		X	49		• 74		S11299	

Nb # S11200 missing

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ARCHIVE SOIL SAMPLE INVENTORY SHEET

9/7/82

BOX SEQUENCE NOS. S11300 - S11399

NUMBER IN BOX 75 (BY COUNT) 78 (BY LOG BOOK)

								SUMMARY
	S113-00	X	25	X	50		- 75	
X	01	X	26		- 51		- 76	
	- 02	X	27		- 52		- 77	
	- 03		- 28		- 53		- 78	
X	04		- 29		- 54		- 79	
X	05	X	30	X	55		- 80	
X	06	X	31		- 56		- 81	
	- 07		- 32		- 57		- 82	
	- 08	X	33		- 58		- 83	
	- 09		- 34		- 59		- 84	
	- 10	X	35		- 60		- 85	
	- 11		- 36		- 61		- 86	
	- 12		- 37		- 62		- 87	
	- 13		- 38		- 63	X	88	
	- 14		- 39		- 64	X	89	
	- 15		- 40		- 65	X	90	
	- 16		- 41		- 66		- 91	
	- 17	X	42	X	67		- 92	
	- 18		- 43	X	68		- 93	
	- 19		- 44	X	69		- 94	
	- 20		- 45		- 70	X	95	
	- 21	X	46		- 71		- 96	
	- 22	X	47	X	72		- 97	
	- 23	X	48		- 73		- 98	
	- 24		- 49		- 74		S11399	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 7/7/82

BOX SEQUENCE NOS. S11400 – S11499

NUMBER IN BOX 74 (BY COUNT) 74 (BY LOG BOOK

								SUMMARY
	S114 00		25		50	X	75	
	01		26		51		76	
	02		27	X	52		77	
	03		28	X	53		78	
	04	X	29	X	54		79	
X	05		30		55		80	
	06	X	31	X	56		81	
	07	X	32		57		82	
	08	X	33		58		83	
X	09		34	X	59		84	
X	10		35		60		85	
X	11		36		61		86	
	12		37		62		87	
	13		38	X	63		88	
	14	X	39		64		89	
X	15		40		65		90	
	16		41	X	66		91	
	17		42		67		92	
	18		43		68		93	
	19		44		69	X	94	
	20		45		70	X	95	
	21		46		71	X	96	
	22		47	X	72	X	97	
X	23		48	X	73		98	
	24		49	X	74		S11499	

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BOX SEQUENCE NOS. S11500 - S11599

NUMBER IN BOX 81 (BY COUNT) 82 (BY LOG BOOK)

										SUMMARY		
	S115 00			• 25			• 50			• 75		
	• 01			• 26			• 51			• 76		
	• 02			• 27			• 52			• 77		
	• 03			• 28			• 53			• 78		
X	04		X	29			• 54		X	79		
	• 05			• 30			• 55			• 80		
	• 06			• 31			• 56			• 81		
	• 07			• 32			• 57		X	82		
	•• 08			• 33			• 58		X	83		
	• 09			• 34	X	59			X	84		
	• 10			• 35			• 60			• 85		
	• 11			• 36	X	61				• 86		
	• 12			• 37	X	62	missing	X	87			
	• 13			• 38	X	63				• 88		
	• 14		X	39			• 64			• 89		
	• 15		X	40		X	65			• 90		
	• 16		X	41			• 66			•• 91		
	• 17		X	42			• 67			• 92		
X	18			• 43			• 68			• 93		
X	19			• 44			• 69			• 94		
X	20			• 45			• 70			• 95		
	• 21			• 46			• 71			• 96		
	• 22			• 47	mr		• 72			• 97		
	• 23			• 48			• 73			• 98		
	• 24			• 49			• 74			S11599		

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/17/82

KMcY

BOX SEQUENCE NOS. S11600 – S11699

NUMBER IN BOX 75 (BY COUNT) 76 (BY LOG BOOK)

									SUMMARY
	S116·00		• 25	X	50			• 75	
	• 01	X	26		• 51	missing	X	76	
	• 02	X	27		• 52			• 77	
	• 03	X	28		• 53			• 78	
X	04		• 29		• 54			• 79	
X	05		• 30	X	55			• 80	
X	06		• 31		• 56			• 81	
X	07	X	32		• 57		X	82	
	• 08		• 33		• 58			• 83	
	• 09		• 34		• 59			• 84	
X	10		• 35		• 60			• 85	
	• 11		• 36		• 61			• 86	
	• 12		• 37		• 62			• 87	
	• 13		• 38		• 63			• 88	
	• 14		• 39		• 64			• 89	
	• 15		• 40	X	65			• 90	
	• 16		• 41		• 66			• 91	
	• 17		• 42		• 67		X	92	
	• 18		• 43		• 68		X	93	
	• 19		• 44	X	69		X	94	
	• 20		• 45	X	70		X	95	
X	21		• 46	X	71			• 96	
	• 22		• 47	X	72			• 97	
	• 23	X	48		• 73			• 98	
	• 24	X	49		• 74			S11699	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82

BOX SEQUENCE NOS. S11700 – S11799 Kimey

NUMBER IN BOX 75 (BY COUNT) 75 (BY LOG BOOK)

								SUMMARY
	S11700		25		50		75	
	01		26	X	51		76	
X	02		27		52		77	
	03		28		53		78	
	04		29	X	54	X	79	
	05		30		55	X	80	
X	06	X	31		56	X	81	
	07		32		57	X	82	
	08		33	X	58		83	
	09		34	X	59		84	
	10		35	X	60	X	85	
X	11	X	36		61		86	
	12	X	37	X	62		87	
	13	X	38		63		88	
X	14		39		64		89	
X	15		40		65		90	
X	16		41		66		91	
	17		42		67		92	
	18		43	X	68		93	
X	19		44		69		94	
	20		45		70	X	95	
	21		46		71		96	
	22		47	X	72		97	
	23		48		73		98	
	24		49		74		S11799	

Kimey

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BOX SEQUENCE NOS. S11800 – S11899 Kimb

NUMBER IN BOX 76 (BY COUNT) 76 (BY LOG BOOK)

								SUMMARY
X	S118 00	X	25		50		75	
	01	X	26		51		76	
X	02		27		52		77	
X	03	X	28		53		78	
X	04		29		54	X	79	
X	05		30		55		80	
	06		31	X	56		81	
	07		32		57		82	
	08		33		58		83	
	09		34		59		84	
	10		35		60		85	
	11		36		61		86	
	12		37		62		87	
	13	X	38		63		88	
	14		39		64		89	
	15		40		65	X	90	
	16		41	X	66	X	91	
	17	X	42		67	X	92	
	18		43	X	68		93	
	19		44	X	69		94	
	20		45	X	70		95	
	21	X	46		71		96	
	22	X	47		72		97	
	23	X	48	X	73		98	
X	24		49		74		S11899	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82
Kimes

BOX SEQUENCE NOS. S11900 – S11999

NUMBER IN BOX 76 (BY COUNT) 78 (BY LOG BOOK)

											SUMMARY
	S119·00			• 25			• 50			• 75	
	• 01			• 26		X	51			• 76	
	• 02			• 27			• 52			• 77	
	• 03			• 28	MISSING	X	53		X	78	
X	04			• 29		X	54		X	79	
	• 05			• 30			• 55		X	80	
	• 06			• 31		X	56			• 81	
	• 07			• 32		X	57			• 82	
	•• 08			• 33		X	58			• 83	
	• 09		X	34			• 59			• 84	
X	10		X	35			• 60			• 85	
	• 11		X	36			• 61			• 86	
X	12		X	37			• 62	MISSING	X	87	
	• 13			• 38			• 63			• 88	
X	14			• 39			• 64		X	89	
X	15			• 40			• 65			• 90	
	• 16			• 41			• 66			• 91	
	• 17			• 42			• 67			• 92	
	• 18			• 43			• 68		X	93	
X	19			• 44			• 69			• 94	
	• 20			• 45			• 70			• 95	
	• 21			• 46			• 71			• 96	
	• 22			• 47		X	72			• 97	
	• 23			• 48			• 73			• 98	
X	24			• 49			• 74			S11999	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82
KWB

BOX SEQUENCE NOS. S12000 – S12099

NUMBER IN BOX ________ (BY COUNT) 77 (BY LOG BOOK)

								SUMMARY
X	S12000		25		50		75	
X	01		26		51		76	
X	02	X	27		52		77	
	03		28		53		78	
	04		29		54		79	
	05		30		55		80	
	06		31		56		81	
	07		32		57		82	
	08	X	33		58		83	
	09		34		59		84	
X	10		35	X	60		85	
	11		36		61		86	
	12		37		62		87	
	13		38		63	X	88	
X	14		39		64	X	89	
	15		40		65	X	90	
	16		41	X	66		91	
	17		42	X	67		92	
	18	X	43	X	68		93	
	19	X	44	X	69		94	
	20	X	45		70		95	
X	21	X	46		71		96	
X	22		47		72		97	
X	23		48		73		98	
X	24		49		74		S12099	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82

KVmdy

BOX SEQUENCE NOS. S12100 - S12199

NUMBER IN BOX 76 (BY COUNT) 76 (BY LOG BOOK)

								SUMMARY
	S12100		25		50	X	75	
	01		26		51	X	76	
	02		27		52	X	77	
	03		28		53		78	
	04		29	X	54	X	79	
X	05		30	X	55		80	
	06		31	X	56		81	
	07	X	32		57		82	
	08	X	33		58		83	
X	09	X	34		59		84	
X	10		35		60		85	
X	11		36		61		86	
X	12		37		62		87	
	13		38		63	X	88	
	14		39		64		89	
	15		40		65		90	
	16	X	41		66		91	
	17		42		67	X	92	
	18		43		68		93	
	19		44		69		94	
	20		45	X	70		95	
X	21		46		71		96	
	22		47		72	X	97	
	23	X	48		73	X	98	
	24		49		74	X	S12199	

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ARCHIVE SOIL SAMPLE INVENTORY SHEE 1 9/1/82

BOX SEQUENCE NOS. S12200 – S12299

NUMBER IN BOX ________ (BY COUNT) 85 (BY LOG BOOK)

								SUMMARY
	S12200		25		50		75	
	01		26		51		76	
	02		27		52		77	
	03		28		53		78	
	04		29		54		79	
	05		30		55		80	
	06		31		56		81	
	07	X	32		57		82	
	08		33		58		83	
	09		34		59		84	
	10		35		60	X	85	
	11		36		61	X	86	
	12		37		62	X	87	
	13		38	X	63	X	88	
	14		39	X	64		89	
X	15	X	40	X	65		90	
	16	X	41		66		91	
	17	X	42		67		92	
	18		43		68		93	
X	19		44		69		94	
X	20		45		70		95	
X	21		46		71		96	
	22		47		72		97	
	23		48		73		98	
	24		49		74		S12299	

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KENNETH W. BROWN (702) 798-2214

ARCHIVE SOIL SAMPLE INVENTORY SHEET 1 4/7/86

BOX SEQUENCE NOS. S12300 - S12399

NUMBER IN BOX ________ (BY COUNT) 86 (BY LOG BOOK)

								SUMMARY
	S12300		25		50		75	
	01		26		51		76	
	02		27		52		77	
	03		28		53	X	78	
	04		29		54	X	79	
	05		30		55	X	80	
	06		31	X	56		81	
	07	X	32	X	57		82	
	08	X	33	X	58		83	
X	09	X	34		59		84	
X	10		35		60		85	
X	11		36		61	X	86	
X	12		37		62		87	
	13		38		63		88	
	14		39		64		89	
	15		40		65		90	
	16		41		66		91	
	17		42		67		92	
	18		43		68		93	
	19		44		69		94	
	20		45		70		95	
	21		46		71		96	
	22		47		72		97	
	23		48		73		98	
	24		49		74		S12399	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/1/82

BOX SEQUENCE NOS. S12400 - S12499   KJ/MEG

NUMBER IN BOX ________ (BY COUNT) 85 (BY LOG BOOK)

								SUMMARY
X	S12400	X	25		50		75	
X	01		26		51		76	
X	02		27		52		77	
	03		28		53		78	
	04		29		54		79	
	05		30		55		80	
	06		31		56		81	
	07		32		57		82	
	08		33		58		83	
	09		34		59		84	
	10		35		60		85	
	11		36		61		86	
	12		37		62		87	
	13		38		63		88	
	14		39		64		89	
	15		40		65		90	
	16		41		66		91	
	17		42		67		92	
	18		43		68	X	93	
	19		44	X	69	X	94	
	20		45	X	70	X	95	
	21	X	46	X	71		96	
	22	X	47		72		97	
X	23	X	48		73		98	
X	24		49		74		S12499	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/1/82

BOX SEQUENCE NOS. S12500 – S12599 Kimes

NUMBER IN BOX ________ (BY COUNT) 87 (BY LOG BOOK)

								SUMMARY
	S125 00		25		50		75	
	01	X	26		51		76	
	02		27		52		77	
	03		28		53		78	
	04		29		54		79	
	05		30		55		80	
	06		31		56		81	
	07		32		57		82	
	08		33		58		83	
	09		34		59		84	
	10		35		60		85	
	11		36		61		86	
	12		37		62		87	
	13		38		63		88	
	14		39		64		89	
	15		40		65		90	
	16		41		66		91	
	17		42		67		92	
	18		43		68		93	
	19		44		69	X	94	
X	20		45	X	70	X	95	
X	21	X	46	X	71	X	96	
X	22	X	47	X	72		97	
	23	X	48		73		98	
	24		49		74		S12599	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 11/1[illegible]

BOX SEQUENCE NOS. S12600 - S12699 KWB

NUMBER IN BOX ________ (BY COUNT) 83 (BY LOG BOOK)

								SUMMARY
	S12600		25		50		75	
	01		26		51		76	
	02		27		52	X	77	
	03		28		53		78	
	04		29		54		79	
	05		30		55		80	
	06		31		56		81	
	07		32	X	57		82	
	08		33	X	58	X	83	
	09		34	X	59	X	84	
X	10		35	X	60	X	85	
	11		36	X	61		86	
	12		37		62		87	
	13	X	38		63		88	
	14	X	39		64		89	
	15	X	40		65		90	
	16		41		66		91	
X	17		42		67		92	
X	18		43		68		93	
X	19		44		69		94	
	20	X	45		70		95	
	21		46		71		96	
	22		47		72		97	
	23		48		73		98	
	24		49		74		S12699	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET 9/7/82
KWB

BOX SEQUENCE NOS. S12700 - S12799

NUMBER IN BOX ________ (BY COUNT) 86 (BY LOG BOOK)

								SUMMARY
	S12700		25		50		75	
	01		26		51		76	
	02		27		52		77	
	03	X	28		53	X	78	
	04		29		54	X	79	
	05		30		55	X	80	
	06		31	X	56		81	
X	07	X	32	X	57		82	
X	08	X	33	X	58		83	
X	09	X	34		59		84	
	10		35		60		85	
	11		36		61		86	
	12		37		62		87	
	13		38		63		88	
	14		39		64		89	
	15		40		65		90	
	16		41		66		91	
	17		42		67		92	
	18		43		68	X	93	
	19		44		69		94	
	20		45		70		95	
	21		46		71		96	
	22		47		72		97	
	23		48		73		98	
	24		49		74		S12799	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET

BOX SEQUENCE NOS. S128xx - S12899

NUMBER IN BOX ________ (BY COUNT) ________ (BY LOG BOOK)

								SUMMARY
	S12800		25		50		75	
	01	X	26		51		76	
X	02	X	27		52		77	
X	03	X	28	X	53		78	
X	04		29		54		79	
	05		30		55		80	
	06		31		56		81	
	07		32		57		82	
	08		33		58		83	
	09		34	X	59		84	
	10		35		60		85	
	11		36		61		86	
	12		37		62		87	
	13		38		63		88	
	14		39		64		89	
	15		40		65	X	90	
	16		41		66	X	91	
	17		42		67	X	92	
	18		43	X	68		93	
X	19		44	X	69		94	
	20		45	X	70		95	
	21		46		71		96	
	22	X	47		72		97	
	23	X	48		73		98	
	24	X	49		74		S12899	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET

BOX SEQUENCE NOS. S12800 - S12899

NUMBER IN BOX [illegible] (BY COUNT) 80 (BY LOG BOOK)

								SUMMARY
	S12800		25		50	X	75	
	01	X	26		51		76	
X	02	X	27		52		77	
X	03	X	28	X	53		78	
X	04		29		54	X	79	
	05		30		55		80	
	06		31		56		81	
	07		32		57		82	
	08		33		58		83	
	09		34	X	59		84	
	10		35		60		85	
	11		36		61		86	
	12		37		62		87	
	13		38		63		88	
	14		39		64		89	
	15		40		65	X	90	
	16		41		66	X	91	
	17		42		67	X	92	
	18		43	X	68		93	
X	19		44	X	69		94	
	20		45	X	70		95	
	21		46		71		96	
	22	X	47		72		97	
	23	X	48		73		98	
	24	X	49		74		S12899	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET

BOX SEQUENCE NOS. 12900 - 12999

NUMBER IN BOX 85 (BY COUNT) 85 [illegible] (BY LOG BOOK)

								SUMMARY
	00		25		50		75	
	01		26		51		76	
	02		27		52		77	
	03		28		53		78	
	04		29		54		79	
	05		30		55		80	
	06		31		56		81	
	07		32		57		82	
	08		33		58		83	
	09		34		59		84	
	10		35		60		85	
X	11		36		61		86	
X	12		37		62		87	
X	13		38		63		88	
X	14		39		64	X	89	
	15		40		65	X	90	
	16		41	X	66	X	91	
	17		42	X	67		92	
	18		43	X	68		93	
	19	X	44		69		94	
	20	X	45		70		95	
	21	X	46		71		96	
	22		47		72		97	
	23		48		73		98	
X	24	X	49		74		99	

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ARCHIVE SOIL SAMPLE INVENTORY SHEET

BOX SEQUENCE NOS. 13000—1399

NUMBER IN BOX 53 (BY COUNT) 53 (BY LOG BOOK)

								SUMMARY
●	00	●	25	●	50	☒	75	
●	01	●	26	●	51	☒	76	
●	02	●	27	●	52	☒	77	
●	03	●	28	●	53	☒	78	
●	04	●	29	●	54	☒	79	
●	05	●	30	●	55	☒	80	
●	06	●	31	●	56	☒	81	
●	07	●	32	●	57	☒	82	
●	08	●	33	●	58	☒	83	
●	09	●	34	☒	59	☒ END	84	
●	10	☒	35	☒	60		85	
●	11	☒	36	☒	61		86	
☒	12	☒	37	☒	62		87	
☒	13	☒	38	☒	63		88	
☒	14	☒	39	☒	64		89	
●	15	☒	40	☒	65		90	
●	16	●	41	☒	66		91	
●	17	●	42	●	67		92	
●	18	●	43	●	68		93	
●	19	●	44	●	69		94	
●	20	●	45	☒	70		95	
●	21	●	46	☒	71		96	
●	22	●	47	☒	72		97	
●	23	●	48	☒	73		98	
●	24	●	49	☒	74		99	

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APPENDIX C-2

INHOUSE PROGRAM AUDIT

ESA LABORATORIES INC
43 WIGGINS AVE
BEDFORD MA 01730
617 275 0100

CLIENT IDENTIFICATION FRED BARNES, H&HS, ENV. HLTH

ADDRESS 1500 W. MOCKINGBIRD LANE
DALLAS, TEXAS 75235

ANALYSES REQUESTED

PAGE 3 OF 7

DATE RECEIVED

DATE ANALYZED

DATE REPORTED

LABORATORY ANALYSIS DATA FORM

	NAME ID	CLOCK EMP #	CLIENT DESIRED INFORMATION	REMARKS	RBC	WBC	MCV	HCT	BLOOD LEAD	HGB Gm%	FEP	ZPP
2	LAKETA		22137-05	00081								
12	DETRICK		22426-05	00082								
12	JONATHAN		21238-05	00083								
2	ANGEL		21220-06	00084								
12	CHARLES		20503-05	00085								
12	DANAYESHA		22053-06	00086								
12	LATISHA		22772-05	00087								
12	KENNEY		22129-07	00088								
	VERONICA		22129-09	00089								
12	CURTIS		21626-05	00090								
12	TANJAYETTE		22772-06	00091								
2	BRONSON		21626-06	00092								
2	BRANDON		20487-05	00093								
12	TRAVAROL		20461-06	00094								
12	JUSTIN		20461-05	00095								

BE COMPLETED BY LAB

NUMBER RECEIVED THIS SHIPMENT

MACRO

Bld Pb

HEM

OTHER

MICRO

Dupl.

Single

ANALYSES THIS SHIPMENT

TOTAL HEMATOLOGIES

OTHER

ILE COPY - DO NOT DETACH

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8

Sample Bar Number	Date Collected	Matrix	CoC form Number	Site ID Code No.	Analytical TAG Number	Analytical Laboratory	Date Shipped to LAB	Chain of Custody form No.	Remarks
B00087	7-12-82	Blood		22772-05	00087	ESA	7-14-82	27754	[illegible]
B00088	7-12-82	Blood		22129-07	00088	ESA	7-14-82	27754	Kenney
B00089	7-12-82	Blood		22129-07	00089	ESA	7-14-82	27754	LESCINSKA
B00090	7-12-82	Blood		21626-05	00090	ESA	7-14-82	27754	CURTIN
B00091	7-12-82	Blood		22772-06	00091	ESA	7-14-82	27754	TANZAYE TIE
B00092	7-12-82	Blood		21626-06	00092	ESA	7-14-82	27754	B.CONION
B00093	7-12-82	Blood		20487-05	00093	ESA	7-14-82	27754	B.CANDON
B00094	7-12-82	Blood		20461-06	00094	ESA	7-14-82	27754	TRAVAROL
B00095	7-12-82	Blood		20461-05	00095	ESA	7-14-82	27754	JUSTIN
B00096	7-12-82	Blood		20537-06	00096	ESA	7-14-82	27755	MIESAR
B00097	7-12-82	Blood		20485-05	00097	ESA	7-14-82	27755	CHAUNCEY
B00098	7-12-82	Blood		20867-05	00098	ESA	7-14-82	27755	SMINGUA
B00099	7-12-82	Blood		20032-05	00099	ESA	7-14-82	27755	JOSE
B00100	7-12-82	Blood		21634-05	00100	ESA	7-14-82	27755	Howard
B00101	7-12-82	Blood		22780-06	00101	ESA	7-14-82	27755	~~[illegible]~~
B00102	7-12-82	Blood		21634-06	00102	ESA	7-14-82	27755	~~[illegible]~~ TAMERA
B00103	7-12-82	Blood		20057-06	00103	ESA	7-14-82	27755	~~[illegible]~~ LILLIAN
B00104	7-12-82	Blood		20857-05	00104	ESA	7-14-82	27755	NIKE
B00105	7-12-82	Blood		20040-05	00105	ESA	7-14-82	27755	
B00106	7-12-82	Blood		20040-06	00106	ESA	7-14-82	27755	~~KEVIN~~
B00107	7-12-82	Blood		22954-05	00107	ESA	7-14-82	27755	~~Shawn~~ KEVIN
B00108	7-12-82	Blood		22897-05	00108	ESA	7-14-82	27755	~~[illegible]~~ SHAWN
B00109	7-12-82	Blood		22865-06	00109	ESA	7-14-82	27755	CHEWBUA
B00110	7-12-82	Blood		22871-07	00110	ESA	7-14-82	27755	COREY
B00111	7-12-82	Blood		22863-05	00111	ESA	7-14-82	27756	NABUSTA
B00112	7-12-82	Blood		22871-06	00112	ESA	7-14-82	27756	CHANDUS
B00113	7-12-82	Blood		22947-06	00113	ESA	7-14-82	27756	CLARENCE
B00114	7-12-82	Blood		22830-05	00114	ESA	7-14-82	27756	LEE JR
B00115	7-12-82	Blood		22947-05	00115	ESA	7-14-82	27756	BOBBY

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Environmental Protection Agency

ATTACHMENT 4

CHAIN OF CUSTODY RECORD
QC SAMPLES

Project Code 68-02-3148	Project Name DALLAS LEAD				Analysis	
TO: ANALYTICAL LAB ESA	SAMPLE DESCRIPTION					
Sample Number	Water	Soil	Sediment	Other	LEAD	REMARKS
B00427				BLOOD	☒	
B00428				BLOOD	☒	
B00429				BLOOD	☒	
B00430				BLOOD	☒	
B00431				BLOOD	☒	
B00432				BLOOD	☒	
B00433				BLOOD	☒	
B00434				BLOOD	☒	
B00435				BLOOD	☒	
B00436				BLOOD	☒	
B00437				BLOOD	☒	
B00438				BLOOD	☒	
B00439				BLOOD	☒	

Relinquished by: (Signature)	Date/Time 7-20-84 1015	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Sample Bank by: (Signature)	Date/Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Originator Files

MG-115(6-82)

8/24

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Sample Bank #	Date Collected	Media	Incoming C of C Form Number	Site ID Code No.	Analytical tag Number	Analytical Lab	Date Shipped to Lab	Lab C of C Form Number	Remarks	Sample Bank Outgoing C of C Form #
B00429	7/20/82	BLOOD	2341	72207-06	00397	ESA	7-30-82	27767	SHEDRIENNE	8129
B00430	7/28/82	BLOOD	2324	72264-05	00398	ESA		27767	KEAUNDRA	8124
B00431	7/28/82	BLOOD	2394	13649-06	00399	ESA		27767	Jerrod	8124
B00432	7/28/82	BLOOD	2391	13599-07	00400	ESA		27767	Montrell	8124
B00433	7/28/82	BLOOD	2381	71514-05	00401	ESA		27768	Robert	8124
B00434	7/28/82	BLOOD	2381	71514-06	00402	ESA		27768	Tachina	8124
B00435	7/28/82	BLOOD	2293	71050-05	00403	ESA		27768	Curtis Jr.	8124
B00436	7/28/82	BLOOD	2293	71050-06	00404	ESA		27768	Veronica	8124
B00437	7/28/82	BLOOD	2277	71076-06	00405	ESA		27768	Andric	8124
B00438	7/28/82	BLOOD	2388	71084-06	00406	ESA		27768	Nakora	8124
B00439	7/28/82	BLOOD	2388	71084-05	00407	ESA		27768	Angela	8124
B00440	7/28/82	BLOOD	2385	71555-06	00408	ESA		27768	STACY	8125
B00441	7/28/82	BLOOD	2385	71555-05	00409	ESA		2769	ANTINO	8125
[illegible]442	7/28/82	BLOOD	2383	71555-30-06	0040	ESA		27769	PATRICK	8125
B00443	7/28/82	BLOOD	2267	11270-07	00411	ESA		27769	ADOLFO	8125
B00444	7/28/82	BLOOD	2267	11270-06	00412	ESA		27769	EDUARDO	8125
B00445	7/28/82	BLOOD	2277	10306-05	00413	ESA		27769	ROBERTO	8125
B00446	7/28/82	BLOOD	2262	20354-06	00414	ESA		27769	KARINA	8125
B00447	7/28/82	BLOOD	2262	20354-07	00415	ESA		27769	FRED	8125
B00448	7/28/82	BLOOD	2261	11259-05	00416	ESA		27769	MARIA	8125
B00449	7/28/82	BLOOD	2378	10314-05	00417	ESA		27770	EMMETT	8125
B00450	7/24/82	BLOOD	2264	11288-05	00418	ESA		2770	PATRICIA	8125
B00451	7/28/82	BLOOD	2331	71043-06	00419	ESA		27770	WILLIAM	8125
B00452	7/28/82	BLOOD	2382	71522-06	00420	ESA		27770	LAKESHIA	8125
B00453	7/28/82	BLOOD	2382	71522-07	00421	ESA		2770	ILSHA	8126
B00454	7/28/82	BLOOD	2382	71522-05	00422	ESA		2770	LAWANDA	8126
B00455	7/28/82	BLOOD	2382	71522-08	00423	ESA		27770	LAKISHA	8126
B00456	7/29/82	Blood	2389	71563-07	00424	ESA		27770	michael	8126
B00457	7/29/82	Blood	2389	71563-06	00425	ESA	7-30-82	27850	ADAM	8126

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Dallas Lead Monitoring Project

Analysis Data Reporting Form

DA 68-02-3168 ☐☐-☐☐☐-☐☐☐☐☐-☐

Comp

	Sample Bank Number	Link Number	Medium	Lab. I.D	Lab Received Date Mo	Lab Received Date Da	Lab Received Time Hr	Lab Received Time Mn	Sample Size	Concentration Soil-ug pb/gm Dust-ug pb/cm²	Not Detected	Trace	Qualitative	Confirmation	Site I.D Code Number
1	S00414		SOIL	GCA						28•8					RSR D022
2	S00027		SOIL	GCA						156•					
3	S00031		SOIL	GCA						74•5					
4	S00032		SOIL	GCA						148•					
5	S00033		SOIL	GCA						214•					
6	S00034		SOIL	GCA						344•					
7	S00035		SOIL	GCA						77•6					
8	S00037		SOIL	GCA						83•1					
9	S00039		SOIL	GCA						116•					
10	S00087		SOIL	GCA						575•					
11	S00095		SOIL	GCA						152•					
12	S00092		SOIL	GCA						50•5					
13	S00083		SOIL	GCA						100•					
14	S00077		SOIL	GCA						459•					
15	S00081		SOIL	GCA						792•					
16	S00415		SOIL	GCA						93•0					RSC M13Y
17										•					
18	QB0004									ND•					
19	QS0004									770•					
20	QK0004									24•4					

ND - NON DETECTED

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Dallas Lead Monitoring Project

Analysis Data Reporting Form

DA 68-02-3168 ☐☐-☐☐☐-☐☐☐☐☐-☐

Com

	Sample Bank Number	Link Number	Medium	Lab. I.D	Lab Received Date Mo	Lab Received Date Da.	Lab Received Time Hr.	Lab Received Time Mn	Sample Size	Concentration Soil-ug pb/gm Dust-ug pb/cm²	Not Detected	Trace	Qualitative	Confirmation	Site I.D. Code Number
1	S00416		SOIL	GCA						95•0					RSR M132
2	S00112		SOIL	GCA						2130•					
3	S00084		SOIL	GCA						212•					
4	S00089		SOIL	GCA						342•					
5	S00110		SOIL	GCA						1660•					
6	S00104		SOIL	GCA						51•7					
7	S00086		SOIL	GCA						59•3					
8	S00077		SOIL	GCA						274•					DMC K14
9	S00085		SOIL	GCA						234•					
10	S00076		SOIL	GCA						75•6					DMC K13
11	S00108		SOIL	GCA						163•					
12	S00105		SOIL	GCA						12[illegible]•					
13	S00074		SOIL	GCA						34•5					DMC J12
14	S00075		SOIL	GCA						27•0					DMC K12
15	S00078		SOIL	GCA						200•					DMC L14
16	S00082		SOIL	GCA						595•					
17										•					
18	QS0005									ND•					
19	QS0005									761•					
20	QA0005									317•					

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DA 68-02-3168
(GCA 1-619-080)

Trans 8/20

QC#8

GCA CONTROL NO.
23449-23468
RN: 14

8/20/82 ④

DALLAS LEAD MONITORING PROJECT
SOIL SAMPLE TRAVELER

SEQ NO.	GCA CONTROL NO.	WEIGHT(G)	ABS	%SCALE	DIL	
1	23459	4.69003	.029			59.3
2	23460	4.73557	.107			274
3	23460RX	4.73557	.004			<10
4	23461	4.97024	.096			234
5	23462	5.13692	.033			75.6
6	23463	5.08770	.069			163
7	NBS	4.94039	.032		10 → 100	761
8	23464	5.05367	.052			123
9	23465	5.21796	.016			34.5
10	23466	5.29543	.013			27.0
11	CC	1	.084			106
12	23456SX	5.02015	~~.134~~ 0.144			~~349~~ 358
13	23467	5.00528	.083			209
14	23468	4.54138	~~.019~~/0.0231		10→100	~~427~~ 595
15	23468TR	4.83844	~~.028~~ 0.029		10→100	~~616~~ 711
16	234578+ [illegible]	3.70752	.057	1667 [illegible]	1→100	
17	23449	4.49359	.038			100
18	23450	5.22838	.197			459
19	23451	5.05534	.034		10→100	797
20	RB	1	.000			<0.25
21	23452	4.70574	.037			93.0
22	CC	1	.086			104
23	23453	5.12375	.041			95.0
24	23454 Hold	5.13762	.009	1.09 [illegible]	1→100	~~1820~~ 2150
25	23454X+	5.77862	~~.014~~ 0.0126	1.49 ug/ml	1→100	9570
26	23455	5.29811	.093			312
27	23456	5.02015	.136 .12 ~~.130~~ / 0.141			342
28	23457 REDIG 8/26	5.02674 ~~4.36229~~	~~.070~~ 0.054		~~10→100~~ 1→100	1660
29	23457TR	2.00217	.019		1→100	6530
30	23458	4.89841	.022			61.7
31	EPA	3.70	.029		98%	3.15
32	CC	1	.084			102

FROM G&S Ed MacKinnon TO WEIGHING Alan Peterson DATE 8-13-82

FROM WEIGHING Alan Peterson TO PREP P Maynard DATE 8/16/82

FROM PREP P Maynard TO AA DATE 8/18/82

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1-617-080 7-[illegible]-78

550-177 Dallas Pb

Samples	ABS	wt		
23680 (10→100)	.020			
23578 (st) (10→100)	.030 (709 mg/g)	4.9898	861 105%	
23557 (st) 10→100	.031	4.7807 732	918	
23456 (st) 10→100	~~.039~~ 0.037	4.8323 724	~~1149~~ 109%	1139
23467 (TR)	.076	~~5.0498~~	217	
23456 (TR)	.112	4.8021	336	
23457 10→100	.054	4.6674	1659	
23477	.068		~~215~~ ~~149~~ 161 [1vv]	
23477 (TR)	.037		111	
23477 (st) 10→100	.031 778 mg/g spike		914 ~~1045~~ 109%	

~~Sam~~ Standards

Blank	.000
.5	.003
1	.007
2	.014
5	.032
10	.072
15	.105
20	.136
25	.171

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DA 68-02-3168
GCA 1-619-080

SOIL SAMPLE QUALITY ASSURANCE

Report to: Dr. Stuart C. Black, EMSL-LV
Date Reported: 8/23/82

Distribution: S. J. Simon
QC Report No.: 8

Sample Numbers: (Sequence as analyzed)

1. S00086
2. S00077
3. S00085
4. S00076
5. S00108
6. S00105
7. S00074
8. S00075
9. S00078
10. S00082
11. S00083
12. S00079
13. S00081
14. S00415
15. S00416
16. S00112
17. S00084
18. S00089
19. S00110
20. S00104

Laboratory name: GCA/Technology Division

Laboratory supervisor: K. T. McGregor
Analyst(s) initials: TH

1. Reagent blank: µg/ml ND

2. Calibration control sample: µg/ml

Found 3.15
True 3.20
Source: EMSL-CIN
% recovery 98

3. Spike extract analysis: µg/ml

Sample extract result 1.09
Spike extract result 2.98
Spike added 2.00
Sample No. S00112
% recovery 95

SOIL SAMPLE QUALITY ASSURANCE (continued)

QC Report No. 8

---

4. Residue reextraction analysis: μg/g

Sample No. S00077

Original extraction 274

Residue extraction ND

% originally extracted 100

---

5. Total recoverable analysis: μg/g

Sample No. S00078

Original result 200

"Total" method result 217

Total recoverable analysis: μg/g

Sample No. S00082

Original result 595

"Total" method result 711

---

6. Laboratory control sample analysis: μg/g

Source: NBS SRM 1645, River Sediment

Found 761

True 714

% recovery 106

---

7. Split extract analysis: μg/g

Sample No. S00089

Original extract result 342

Split extract result 338

---

8. Spiked sample analysis: μg/g

Sample No. S00089

Spiked sample result 1131

Unspiked sample result 342

Spike added 724

% recovery 109

APPENDIX M

TECHNICAL PROPOSAL FOR A GEOSTATISTICAL STUDY OF DALLAS LEAD DATA

TECHNICAL PROPOSAL
FOR
A GEOSTATISTICAL STUDY
OF
DALLAS LEAD DATA

STATEMENT OF OBJECTIVES

The objective of this project is to provide the U.S. Environmental Protection Agency Exposure Assessment Division in Las Vegas, Nevada, with a geostatistical analysis of the lead (Pb) contamination surrounding each of three smelters in the Dallas Lead Project. The study will utilize a two-dimensional geostatistical method with two dimensional model representation drawn by computer.

This study is also regarded as an opportunity for the U.S. Environmental Protection Agency Exposure Assessment Division to enhance their understanding of the application of geostatistical techniques to the evaluation of areas contaminated by hazardous wastes. It is the longstanding tradition of Geostat Systems, Inc., and perhaps unique to the geostatistical field, to incorporate on-site training during the execution of the project. However, it is to be understood that this training option, if taken, would increase the duration of the project and its cost.

Also, Geostat Systems, Inc., offers to let the U.S. Environmental Protection Agency Exposure Assessment Division buy the basic geostatistical programs used in the study. The description of the programs and their current price list are given in Appendix 1.

Geostat Systems, Inc. is one of the leading geostatistical firms in the world and as such feels particularly qualified to perform the proposed analysis. Geostat has pioneered a great many of the automatic evaluation techniques for regionalized variables that are available today. The development of these techniques is the result of many years of practical application to a great variety of variables, and this practical experience is invaluable in the interpretation of problems. Geostat is also backed-up by several of the best university research groups worldwide.

STATEMENT OF WORK

The work to be done can be divided in five parts: first, the assemblage, entry and verification of sample data onto the Geostat Systems, Inc. computer facilities; second, a study of the statistical characteristics of the data in each of the three zones and on an overall basis; third, derivation of variogram models in each area; fourth, kriging of data values; and fifth, display of kriged output.

Part 1: Data capture

Data as received from the U.S. Environmental Protection Agency Exposure Asse[illegible] Division in the form of cards, data sheets, or computer tape will be entered and verified on Geostat Systems computer facilities. Data must include at least X-Y coordinates and a sample value. It should be noted that manual entry of data by Geostat Systems (data sheets) will be the most expensive option, and as such is the option quoted here. Expected time: 4 days.

Part 2: Statistical analysis

Histograms and basic statistics will be computed for each of the three areas of interest to determine basic parameters. Histograms and basic statistics will also be calculated on an overall basis for comparison. Expected time: 2 days.

Part 3: Structural analysis

Variograms of sample values in each of the three regions will be computed. The variograms are calculated along several directions to check for anisotropies. Overall variograms will also be computed for comparison purposes. The variogram features are interpreted in geologic terms, and models are fitted to the experimental variograms. Expected time: 6 days.

Part 4: Kriging

Point kriging of sample parameters will be performed on a regular grid in each area of the study. This method takes into account the continuity of sample data as expressed by their variograms, the location of the samples around the point, and their clustering. The level of contaminant present is estimated at each point and a precision value is given for each estimate. Expected time: 4 days.

Part 5: Display

Kriging results will be displayed as contour maps at estimated contaminant levels for all areas of the study. Accompanying these maps will be contour maps in each area of the kriged error maps (precision maps) indicating the confidence with which the estimation was made. Expected time: 2 days.

Estimates of time set forth under this statement of work follow the assumption that a representative of the U.S. Environmental Protection Agency Exposure Assessment Division will be present during the structural analysis and kriging phases of the study. Itemized estimates of time for studies both with and without a representative of your staff are outlined in the following cost schedule.

APPENDIX 1

THE TOXIPAC PACKAGE

THE TOXIPAC PACKAGE

INTRODUCTION

This is our basic environmental package and has been designed for hydrogeologic problems including aquifer modelling, pollutant contamination assessment, and soils analysis.

Functions include: two- or three-dimensional variogram computation, variogram model verification and optimization, interactive definition of kriging parameters and determination of optimum sampling patterns, two- or three-dimensionsal kriging, gridding of irregularly spaced data with universal kriging and the option of automatic definition of the continuity structure (generalized covariance), and the contouring of data.

Options include complete conditional simulation capabilites featuring data normalization, simulation, conditionalization, and distribution restoration. Other options allow block display on either printer or plotter, standard statistical analysis, and rotation of original coordinate system.

All the programs of the package are written in standard FORTRAN. We have tested them on over a hundred projects and they are presently running on IBM, AMDHAL, CDC, DEC, UNIVAC, BURROUGHS, VAX and PRIME systems. The entire package is about 17,000 cards of code.

Convenience of use and optimization of computer time are the two factors that we considered most. As an example, all the array and vector space is defined through a single vector with subsequent use of pointers. The dimension of this unique vector is defined in a driver main program. This eliminates the need for recompiling the program when the dimension must be changed. Also when the dimension is exceeded, a message with the necessary dimension is printed before execution is terminated.

A summary description and examples of output of the main programs in the package follows. A more detailed documentation and the current price list are available on request.

MARECX

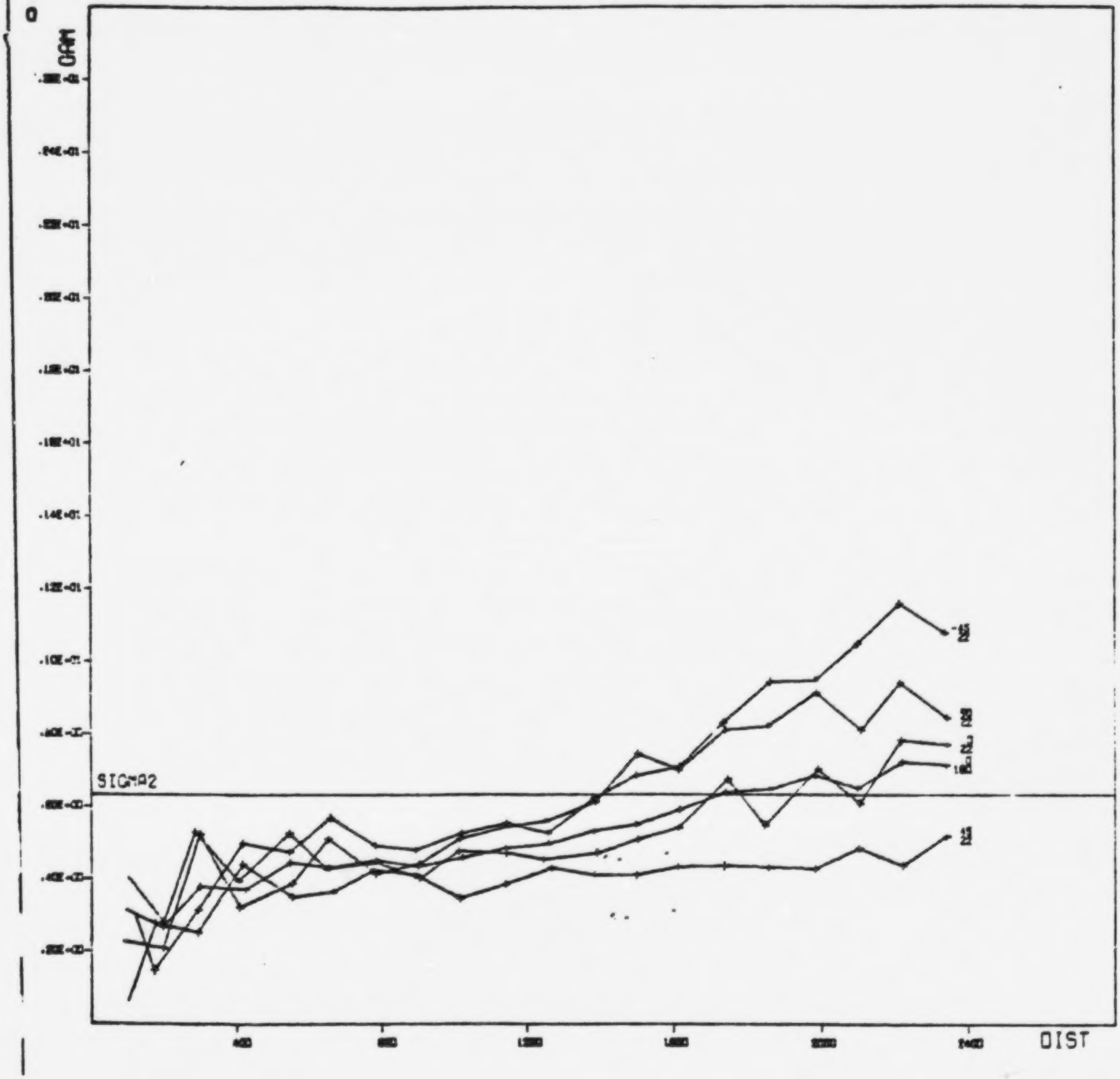
All areas of interest differ geologically, and you should not attempt to estimate them in the same way as you would by inverse distance, inverse square distance methods, or other arbitrary interpolation techniques.

The particular properties of a given area such as similarities between close samples, the zone of influence of a sample, the quick change of values on short distance, anisotropy in the variable and, more generally, continuity of the variable for a given deposit, is reflected by the VARIOGRAM.

The variogram is the basis of each geostatistical estimation. Its essential function is to test the accuracy of different estimation methods.

MARECX can compute, print and plot absolute, relative and logarithmic variograms on irregularly gridded data, in two or three dimensions, for several variables in as many as five directions in a single run. You can look at directions oblique to the plane of coordinates through the preprocessor program ROT.

Several options are opened to you in order to specify limits on variables or coordinates, the directions of variograms to be computed, and which are to be printed and/or plotted.



VARIABLE CUGR GENERAL RELATIVE VARIOGRAM
TEST DEPOSIT - PROGRAM MARECX (HORIZONTAL VARIOGRAPHY)

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KRIJAC

The variogram is the single most important tool of geostatistics. Unfortunately the determination of an experimental variogram and the subsequent modeling are two steps that open the door to subjective decisions. The best variogram is not the one that will "fit the curve" the most nicely but rather the one that will produce, during kriging, the smallest nonbiased estimate errors.

Program KRIJAC was designed to test a variogram model by computing the errors involved by kriging control points (data points) with the surrounding ones. Statistics and histograms of the ensuing errors enable the user to objectively compare vairogram models in order to select the "best" model.

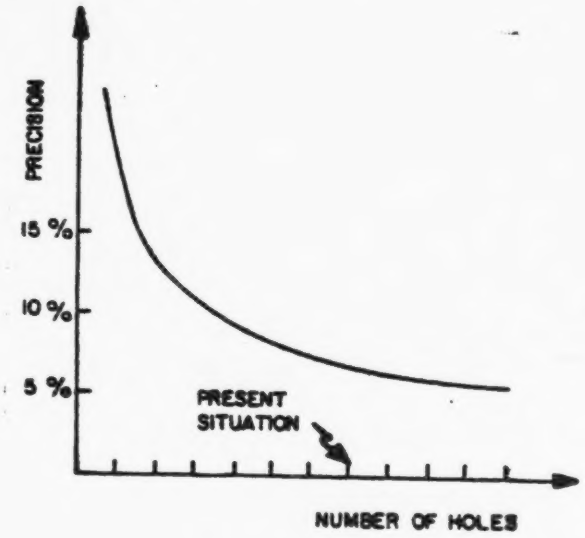
Several options enable the user to control the location and extent of the test area, the size of the kriging matrix and the type of variogram model. Facilities are also provided that allow the use at run time of a user-written variogram function which thus enables one to test virtually any variogram model.

Outputs of the program are:

- Echo printing of selected options
- Echo printing of input records
- Printout of kriged estimates and errors
- Statistics and histograms on errors

KRIVAR

KRIVAR is a powerful, user friendly, interactive kriging program which allows the user to determine the sufficiency of pre-existing data before more samples are taken or additional wells are placed. Once the variogram is obtained, KRIVAR uses this model along with the data to determine the best location to sample next by locating the spot that yields the greatest reduction in the variance, or in other words, the greatest increase in confidence. Thus, KRIVAR provides an objective as well as cost effective way of planning and/or justifying additional sampling that will fall within specified budget or level of safety constraints.



The KRIVAR Program is divided into two distinct phases:

1. A primary phase, in which you specify the intial value of certain parameters; and

2. A secondary, interactive, phase in which you can add, replace, or eliminate some samples and/or modify the parameters set in the primary phase.

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```
ENTER COMMAND & SAMPLE COORDINATES
>a 1 0.0 0.0 0.0
>a 2 50.0 50.0 0.0
>k


NSAMP    KRIGING VAR.     KRIGING STD.
   2      .7163331E-01      .2676440E+00

ENTER COMMAND & SAMPLE COORDINATES
>k 1

POINT       X         Y          Z         WEIGHT
   1       0.00      0.00       0.00     .6117E+00
   2      50.00     50.00       0.00     .3883E+00
NSAMP    KRIGING VAR.     KRIGING STD.
   2      .7163331E-01      .2676440E+00

ENTER COMMAND & SAMPLE COORDINATES
>d


+++ WARNING +++ ASKED TO DELETE ENTIRE LIST
+++ CONFIRM WITH A 'D' OR HIT RETURN KEY TO CANCEL
>d

ENTER COMMAND & SAMPLE COORDINATES
>s

ENTER NUMBER OF GRID POINTS IN X,Y,Z
>2 2 1

ENTER X,Y,Z COOR. OF LOWEST SOUTH-WEST GRID POINT
>-50 -50 0

ENTER X,Y,Z GRID SIZE
>100 100.0 0

ENTER COMMAND & SAMPLE COORDINATES
>k 1

POINT      X          Y          Z         WEIGHT
   1     -50.00     -50.00      0.00      .2500E+00
   2     -50.00      50.00      0.00      .2500E+00
   3      50.00     -50.00      0.00      .2500E+00
   4      50.00      50.00      0.00      .2500E+00
NSAMP    KRIGING VAR.     KRIGING STD.
   4      .4179059E-01     .2044275E+00

ENTER COMMAND & SAMPLE COORDINATES
>f


+++ ASKED TO TERMINATE EXECUTION
CONFIRM WITH A 'E' OR HIT RETURN KEY TO CANCEL
>e

NORMAL END OF PROGRAM
```

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KRIGE3A

This is the core of the estimation package, i.e. where the optimal estimation of points or blocks from nearby samples is done. In statistical terms, optimality means no systematic errors and the smallest possible error. Kriging meets these requirements because:

- it uses the variogram which reflects the particular continuity of the variables in your study area (this is an advantage over all other estimation methods)

- it uses all the surrounding samples

- it considers the proper geometric relations between the samples and the blocks (better than the inverse or the inverse square distance method)

- it considers the geometric relations between the samples themselves (another advantage over all other estimation methods).

KRIGE3A is a kriging program designed for the estimation a large three dimensional block models as well as for points. Computer time is reduced to a minimum through the use of efficient search algorithms. With this program, you can do regular kriging with an absolute or relative variogram or lognormal kriging. You can also make use of the sample search ellipsoid with axes possibly oblique to the coordinate axes.

KRIGE3A produces a <block file> with estimated values and associated standard errors. This file is accepted by the contouring program ISOPOLY; or for blocks may be processed through either BLKMAP or BLKPLT for level or section maps.

```
11    22    4 11  .13259E+00   .26236E+00
12    22    4 11  .22521E+00   .25475E+00
13    22    4 14  .37532E+00   .25807E+00
14    22    4  8  .83474E+00   .36310E+00
15    22    4 11  .36110E+00   .28147E+00
16    22    4 11  .23239E+00   .28753E+00
17    22    4 13  .30266E+00   .26271E+00
18    22    4  8  .36952E+00   .38961E+00
20    22    4 10  .24770E+00   .35629E+00
11    23    4 14  .16840E+00   .26980E+00
12    23    4  9  .22707E+00   .27597E+00
13    23    4  9  .56981E+00   .30244E+00
14    23    4  8  .85253E+00   .29808E+00
15    23    4  9  .32655E+00   .29788E+00
16    23    4  6  .27601E+00   .40345E+00
17    23    4  8  .35173E+00   .30390E+00
18    23    4  6  .31924E+00   .37879E+00
19    23    4  3  .22579E+00   .53831E+00
20    23    4  5  .20363E+00   .43647E+00
11    24    4 11  .17373E+00   .29380E+00
12    24    4  8  .27257E+00   .29710E+00
13    24    4  5  .44051E+00   .31052E+00
14    24    4  8  .54081E+00   .28664E+00
15    24    4 10  .38981E+00   .29566E+00
16    24    4  8  .28249E+00   .29245E+00
17    24    4  8  .31630E+00   .28154E+00
18    24    4  7  .26440E+00   .27097E+00
19    24    4  8  .24266E+00   .28176E+00
20    24    4  8  .22110E+00   .32772E+00
11    25    4  9  .34792E+00   .30309E+00
12    25    4  9  .58371E+00   .29408E+00
13    25    4  6  .59781E+00   .30203E+00
14    25    4 11  .51164E+00   .29413E+00
15    25    4  9  .33272E+00   .30917E+00
16    25    4  9  .32808E+00   .31155E+00
17    25    4 11  .34571E+00   .29512E+00
18    25    4  8  .24074E+00   .30297E+00
19    25    4 11  .22960E+00   .28895E+00
20    25    4  9  .28129E+00   .30258E+00

+++++ KRIGE3A V1.6 —— PERFORMANCE SUMMARY +++++

TOTAL NUMBER OF BLOCKS         :    100
TOTAL NUMBER OF KRIGED BLOCKS  :     98
NUMBER OF SAMPLES READ         :   2086
NUMBER OF SAMPLES DELETED      :   1656
NUMBER OF SAMPLES USED         :    430
NO ERRORS WERE DETECTED
***** NORMAL END OF PROGRAM *****
```

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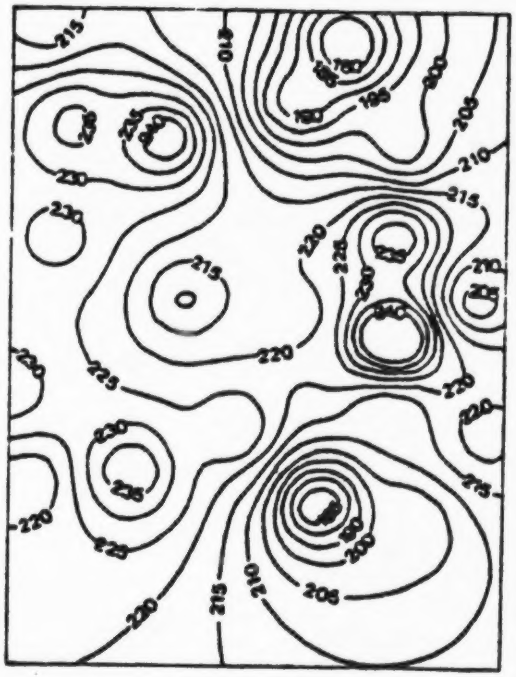
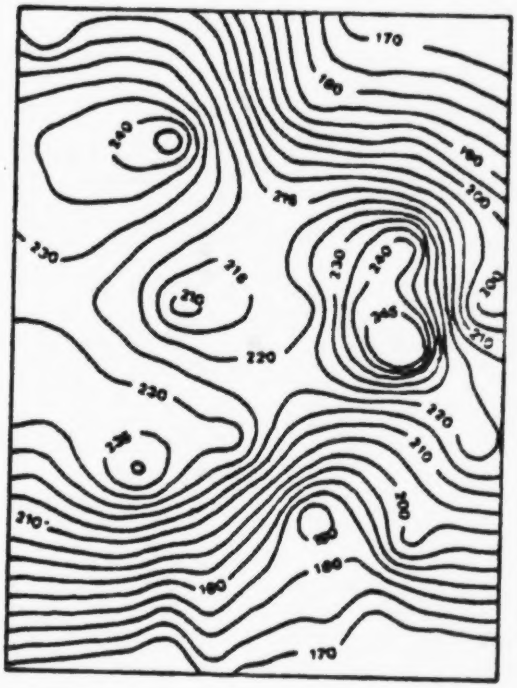
Why the need for a new contouring package?

The examples below show maps of oil shale data. One was produced by using an inverse squared distance weighting method, the other was produced by kriging. Can you see the difference


215
210
180
185
180
195
200
205
235
235
240
230
210
215
230
220
225
235
230
240
210
205
225
215
220
220
230
235
220
225
220
215
210
205
200
190


170
180
240
190
215
200
230
215
210
230
240
245
200
210
220
230
220
210
235
200
210
190
180
180
170

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COVPAC

All by itself, COVPAC is like a complete geostatistical estimation package. It features an optimal interpolation method based on the theory of generalized covariances. Starting from a file of irregularly gridded data in two dimensions, the program does a statistical analysis of the data, then tests for the presence of any trend in the data and determines the most likely spatial covariance. This is followed by the kriging of point or block values on a regular grid. The output file contains the estimated value at each grid point or block with the associated standard error. Isoline maps of the value itself or the standard error can then be produced by the ISOPOLY program.

An interesting feature of COVPAC is that the order of the trend and the spatial covariance are determined by testing the interpolation method on the original data points themselves. They are removed one by one and estimated with the remaining points. The program selects the model which gives the smallest average error and a correct theoretical estimate of the error.

Options include the possibility for the user to bypass the automatic definition of the trend and spatial covariance and introduce his own variogram model.

```
*****************************************
*                                       *
*        TESTING THE MODELS OF THE      *
*  GENERALIZED COVARIANCE FOR THE CASE :*
*                                       *
*          K=1---LINEAR  DRIFT          *
*                                       *
*****************************************

              MODEL NUMBER 4
              -------------

     PARAMETERS OF THE GENERALIZED COVARIANCE
 CO =  .25710E+00  C1 = -.57599E-02  C2 = 0.




  STATISTICS OVER THE      I                     I OVER POINTS KRIGED I OVER POINTS WHOSE
      DISTRIBUTION         I OVER ALL POINTS     I WITH MORE THAN 12  I EXPERIMENTAL ERROR
  OF THE EXPERIMENTAL      I                     I      SAMPLES       I    IS IN A GIVEN
        ERRORS             I                     I                    I      INTERVAL
---------------------------+---------------------+--------------------+--------------------
AVERAGE RESIDUAL ERROR     I       .67207E-01    I     .67207E-01     I      .32220E-01
AVERAGE ABSOLUTE ERROR     I       .60485E+00    I     .60485E+00     I      .50906E+00
AVERAGE SUM OF SQUARES     I       .60156E+00    I     .60156E+00     I      .39788E+00
AVERAGE THEORETICAL ERROR  I       .36007E+00    I     .36007E+00     I      .35107E+00
AVERAGE ERROR RATIO        I       .20383E+01    I     .20383E+01     I      .15928E+01
ADDED ERRORS RATIO         I       .16707E+01    I     .16707E+01     I      .11333E+01


                     HISTOGRAM OF EXPERIMENTAL ERRORS
                     --------------------------------

                     MEAN               =  .67207E-01
                     STANDARD DEVIATION =  .77268E+00

+------------------------+-----+------------------------------------------------------+
I   FROM        TO       I  N  I                    FREQUENCY                         I
+------------------------+-----+------------------------------------------------------+
I -.15E+01   -.13E+01    I   1 I *****                                                I
I -.13E+01   -.12E+01    I   1 I *****                                                I
I -.12E+01   -.10E+01    I   1 I *****                                                I
I -.10E+01   -.86E+00    I   2 I **********                                           I
I -.86E+00   -.71E+00    I   5 I *************************                            I
I -.71E+00   -.55E+00    I   3 I ***************                                      I
I -.55E+00   -.40E+00    I   7 I ***********************************                  I
I -.40E+00   -.24E+00    I   8 I ****************************************             I
I -.24E+00   -.87E-01    I   8 I ****************************************             I
I -.87E-01    .67E-01    I   6 I ******************************                       I
I  .67E-01    .22E+00    I   9 I *********************************************        I
I  .22E+00    .38E+00    I   3 I ***************                                      I
I  .38E+00    .53E+00    I   3 I ***************                                      I
I  .53E+00    .69E+00    I   0 I *                                                    I
I  .69E+00    .84E+00    I   0 I *                                                    I
I  .84E+00    .99E+00    I   1 I *****                                                I
I  .99E+00    .11E+01    I   0 I *                                                    I
I  .11E+01    .13E+01    I   0 I *                                                    I
I  .13E+01    .15E+01    I   0 I *                                                    I
I  .15E+01    .16E+01    I   0 I *                                                    I
+------------------------+-----+------------------------------------------------------+
I   FROM        TO       I  N  I                    FREQUENCY                         I
+------------------------+-----+------------------------------------------------------+
```

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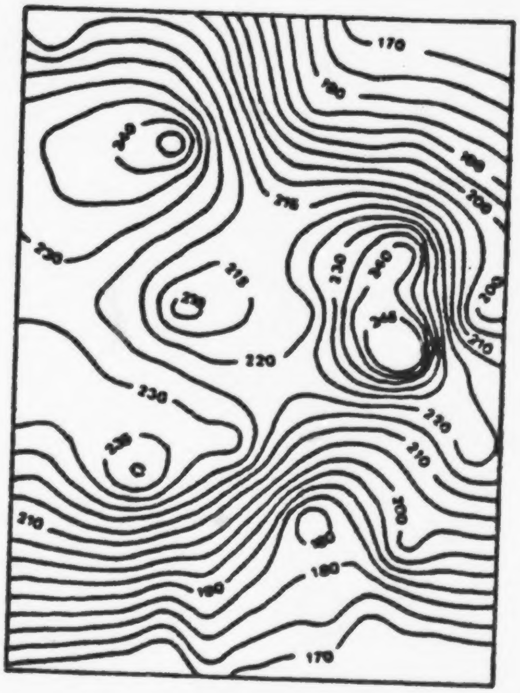
ISOPOLY

ISOPOLY takes the optimally interpolated grid produced by COVPAC (or any other gridding/blocking program) and it fits a bicubic spline surface to the grid values. The isolines are then traced from this surface. As can be seen from the example, the contours are of extremely good quality and virtually free from kinks.

The program includes a multitude of options such as the choice of up to five different ranges between contours, the feathering of contours in regions of high gradient, a metric/engligh scale selectr, and an automatic blanking of contours in regions of poor precision.

The program can also interface the file of original control points and plots them with their associated measured value on the contour map.

The size of the contour maps drawn by ISOPOLY is only limited by the size of the plotting equipment. However if you are not lucky enough to work on a large flat bed plotter, you can get maps of 40 inches size on a small 10 inches plotter because the program automatically splits large maps in bands which can be later assembled.


170
180
240
190
215
200
230
215
230
230
240
200
245
210
220
230
220
230
210
200
210
190
180
190
170

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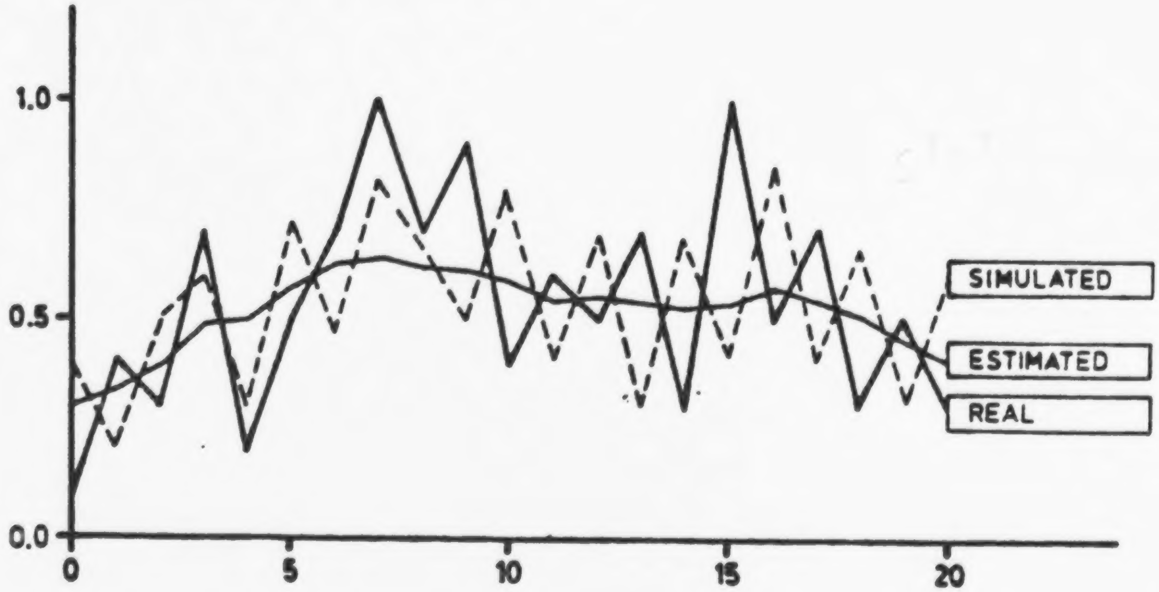
INTRODUCTION

POLYSIM is a simulation package - Simulation is different from estimation. When you estimate points, you want to minimize the average difference between your estimates and the real values. This is the best fit line concept. While kriging is an optimal estimation procedure, it cannot fill in the details that have not been measured. In the case where values are changing rapidly over short distances, you end up with very smooth estimates that go through the middle of the highs and the lows of your real data. Then, the variability of your estimates is much lower than the variability of your real values.



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Simulation aims at reproducing the variability, continuity and distribution of transmissivity, hydraulic head, porosity or level of pollutant contamination in an aquifer. This means that the variance, variogram, and histogram of the simulated values are the same as the variance, variogram and histogram of the real measured values. Thus, simulation is a very powerful technique to reproduce in a very realistic way many possible versions of reality.

The package is made of four programs: TGAUSS does an analysis of the histogram of the sample data and ITGAUSS makes sure that the simulated data are distributed according to this histogram. SIMUL simulates data with a given variogram through the turning bands method. Two different programs are used depending of the dimensionality of the data: SIMUL2 for 2-D data and SIMUL3 for 3-D data. Finally, it is also possible to have the simulated values generated around sample points looking like the values at these points. This operation is called conditionalization. It is performed by the SIMUL2C (2-D data) or SIMUL3C (3-D data) programs.

All the programs of the package are written in standard FORTRAN. They are presently running on IBM, CDC and DEC systems. The entire package is about 5000 cards of code.

Convenience of use and optimization of computer time are the two factors that we considered most. As an example, all the arrays and vector space is defined through a single vector with subsequent use of pointers. The dimension of this unique vector is defined in a driver main program. This eliminates the need for recompiling the program when the dimension must be changed. Also, when the dimension is exceeded, a message with the necessary dimension is printed before execution is terminated.

A summary description and examples of output of the main programs in the package follows. A more detailed documentation and the current price list are available on request.

TGAUSS

When simulation is aimed at generating data with the same histogram and variogram as available sample data, a first step is to analyze the sample data. TGAUSS examines their histogram and finds a model to represent it.

The model is a function which relates the histogram of the sample data to the histogram of standard gaussian data. For example, if data are lognormal this function is exponential. Because the program might also be used for disjunctive kriging, the function is defined as an expansion of Hermite polynomials of the gaussian deviates.

Output of the program includes:

+ a standard statistical analysis of the sample data
+ the definition of the normalization function
+ an analysis of the goodness of fit of this function
+ a file of «normalized» sample data
+ a standard statistical analysis of the «normalized» sample data
+ a simulation of the histogram of the sample data from gaussian deviates using the model.

The next step of the simulation procedure when you start from sample data is to analyze the variograms of the «normalized» sample data through the MARECX program of the TOXIPAC PACKAGE.

```
*** DATANW RUN *** NORMALIZATION OF DATA ***

                    NORMALIZATION FUNCTION

              EXPANSION WITH  4 HERMITE POLYNOMIALS

                HERMITE POLYNOMIALS COEFFICIENTS

                        DEG.    VALUE

                         0 -.137300E+01
                         1  .739756E+00
                         2 -.382757E-01
                         3  .109534E-01

              THEORETICAL ST DEVIATION =    .742E+00

                 GOODNESS OF FIT OF THE MODEL

                 X-VALUE    Y-EXPER    Y-THEOR

               -.313E+01 -.461E+01 -.425E+01
               -.281E+01 -.461E+01 -.387E+01
               -.250E+01 -.351E+01 -.351E+01
               -.219E+01 -.322E+01 -.318E+01
               -.188E+01 -.281E+01 -.287E+01
               -.156E+01 -.253E+01 -.258E+01
               -.125E+01 -.230E+01 -.230E+01
               -.938E+00 -.204E+01 -.204E+01
               -.625E+00 -.183E+01 -.179E+01
               -.313E+00 -.161E+01 -.156E+01
               0.        -.131E+01 -.133E+01
                .313E+00 -.108E+01 -.112E+01
                .625E+00 -.892E+00 -.905E+00
                .938E+00 -.713E+00 -.696E+00
                .125E+01 -.528E+00 -.489E+00
                .156E+01 -.301E+00 -.281E+00
                .188E+01 -.513E-01 -.709E-01
                .219E+01  .223E+00  .144E+00
                .250E+01  .419E+00  .365E+00
                .281E+01  .708E+00  .596E+00
                .313E+01  .788E+00  .836E+00
```

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SIMUL2 (3)

This is the basic program of the package. It produces simulated values of a gaussian deviate with a given variogram either on a fixed grid or at points with a specific location.

The variogram to reproduce is any combination of nugget effect, spherical, exponential or gaussian components. Any kind of 2D or 3D anisotropy, geometrical or zonal, is accomodated.

A statistical analysis of the distribution of the simulated values both at sample and grid points, is a standard feature of both programs. In addition, for the SIMUL2 program you can ask for the computation of the variograms along the four principal directions of the grid and a printer map of the simulated grid points.

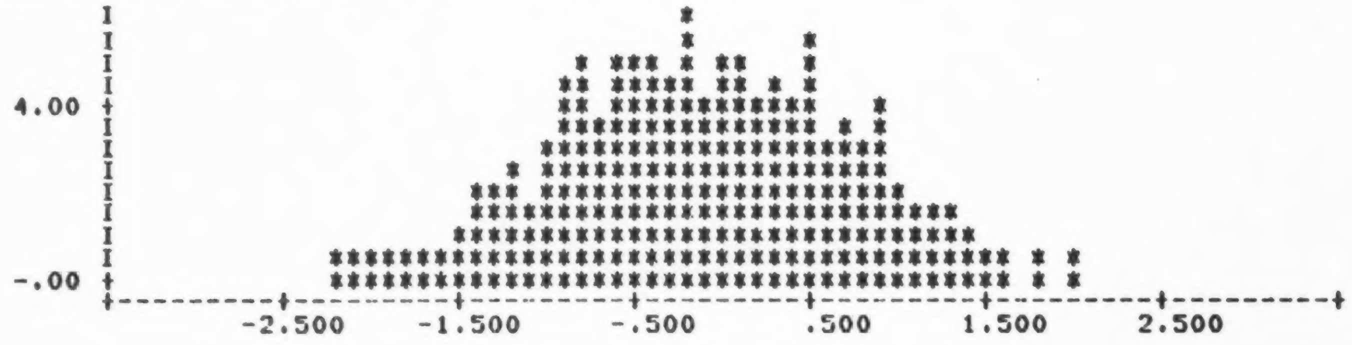
When the program is used with sample points, the output file of normalized sample data of TGAUSS is needed. In that case the program produces two files of simulated values, one for the sample points and the other for the grid points. The two files can then be used in the «conditionalization» programs. When simulation is not dependent of sample data, there is no input file needed and only one output file of simulated grid points is produced.

```
**** UNCONDITIONAL SIMULATION ****

SIMULATED VALUES AT GRID POINTS
NUMBER OF VALUES   =     3200
AVERAGE            =   .25262E-01
STANDARD DEVIATION =   .83830E+00
MINIMUM            =  -.20999E+01
MAXIMUM            =   .25200E+01
```

```
**** UNCONDITIONAL SIMULATION ****

HISTOGRAM OF SIMULATED VALUES AT GRID POINTS
```



SIMUL2C (3C)

This program performs a «conditionalization» of the random normal deviates produced by SIMUL2 (3) program. In simple terms, this «conditionalization» consists in shifting the original «normalized» values in such a way as to produce grid values that resemble closely sample values. The method uses kriging as an exact interpolation procedure of the grid points from the sample points.

Thus three different sets of values are required to the program:

- Normalized real data at sample points (output of TGAUSS)
- Simulated values at sample points (output of SIMUL2 (3))
- Simulated values at grid points (output of SIMUL2 (3))

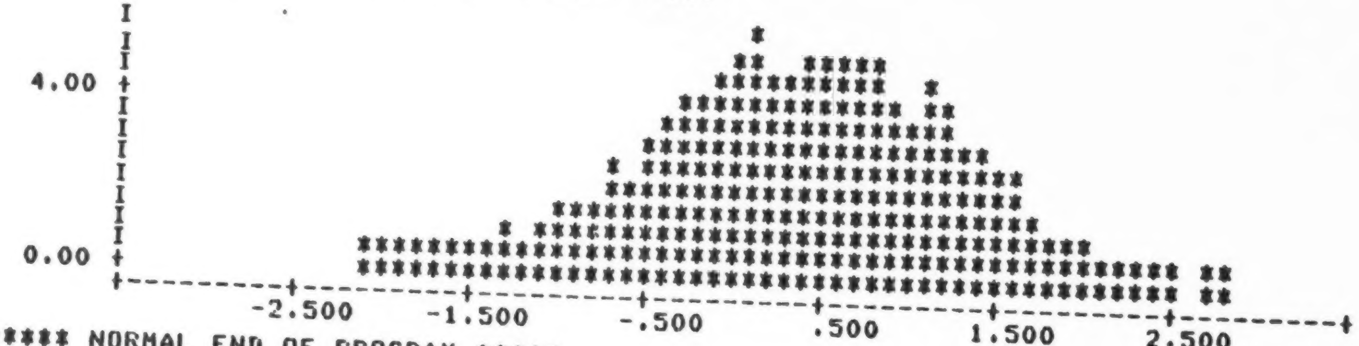
The conditionalization may be restricted to a specific area of the grid, and the variogram function is also user-defined in the same manner as in SIMUL2 (3). You can also choose the parameters of the ellipsoid search zone to use when kriging the grid points from the sample points.

A statistical analysis of the distribution of the simulated values both at sample and grid points, is a standard feature of both programs. In addition, for the SIMUL2C program, you can ask for the computation of the variograms along the four principal directions of the grid and a printer map of the simulated grid points.

```
**** TEST RUN **** CONDITIONAL SIMULATION ****
***** STATISTICS ON SIMULATED GRID POINTS *****
NUMBER OF GRID POINTS SIMULATED =     3200
MINIMUM                         =   -2.06077
MAXIMUM                         =    2.87940
AVERAGE                         =     .44737
STANDARD DEVIATION              =     .77884



**** TEST RUN **** CONDITIONAL SIMULATION ****
***** HISTOGRAM OF SIMULATED VALUES *****
```



```
***** NORMAL END OF PROGRAM *****
```

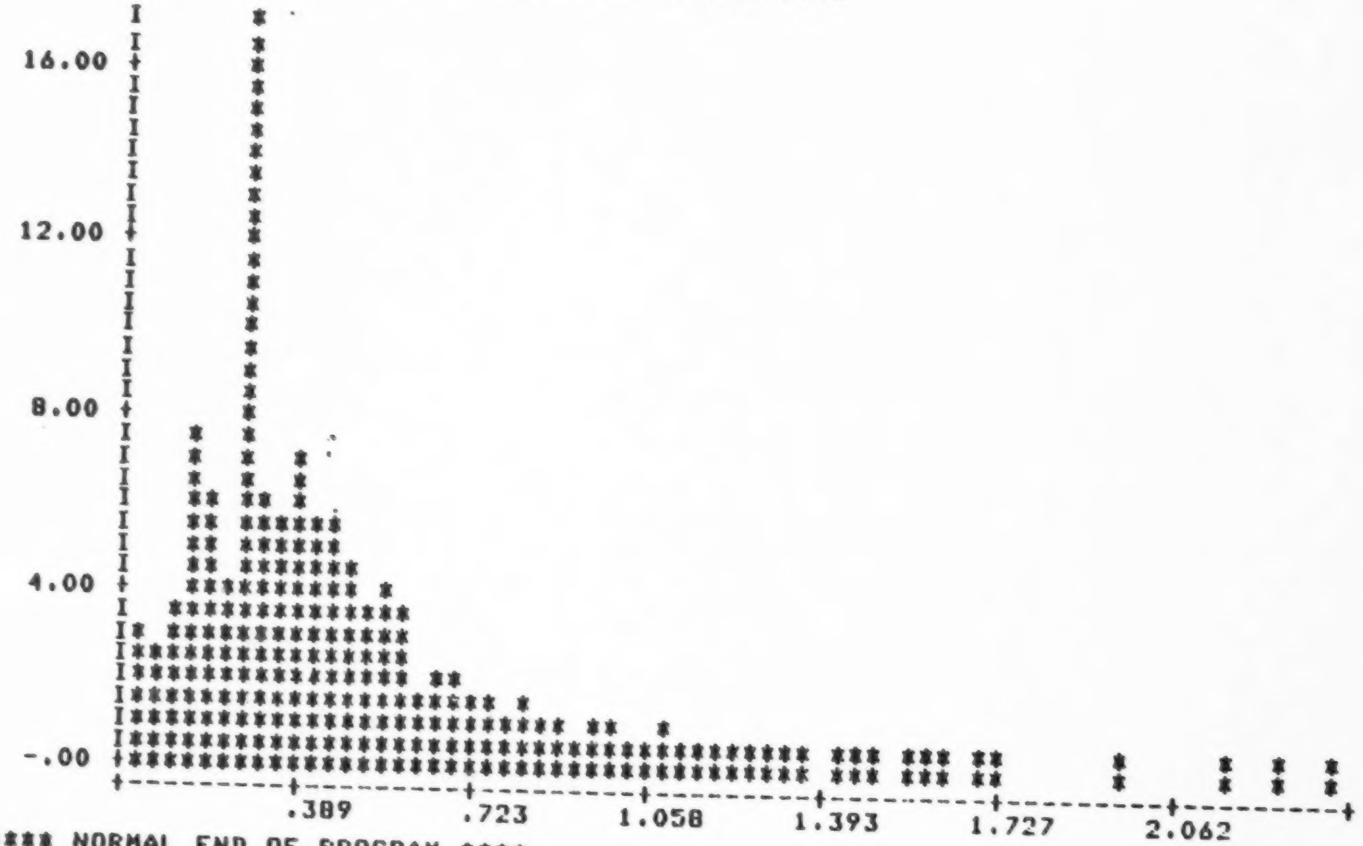
ITGAUSS

This is the last step in the chain of the simulation programs. It transforms simulated normal values produced by SIMUL2 (3) or SIMUL2C (3C) in such a way that the transformed values are distributed according to a specific histogram.

The model can be a normal or a lognormal model with a given mean and variance. More generally, it can be defined by the expansion of a transformation function with Hermite polynomials. This expansion can be derived by the program TGAUSS from experimental data distributed according to the required model.

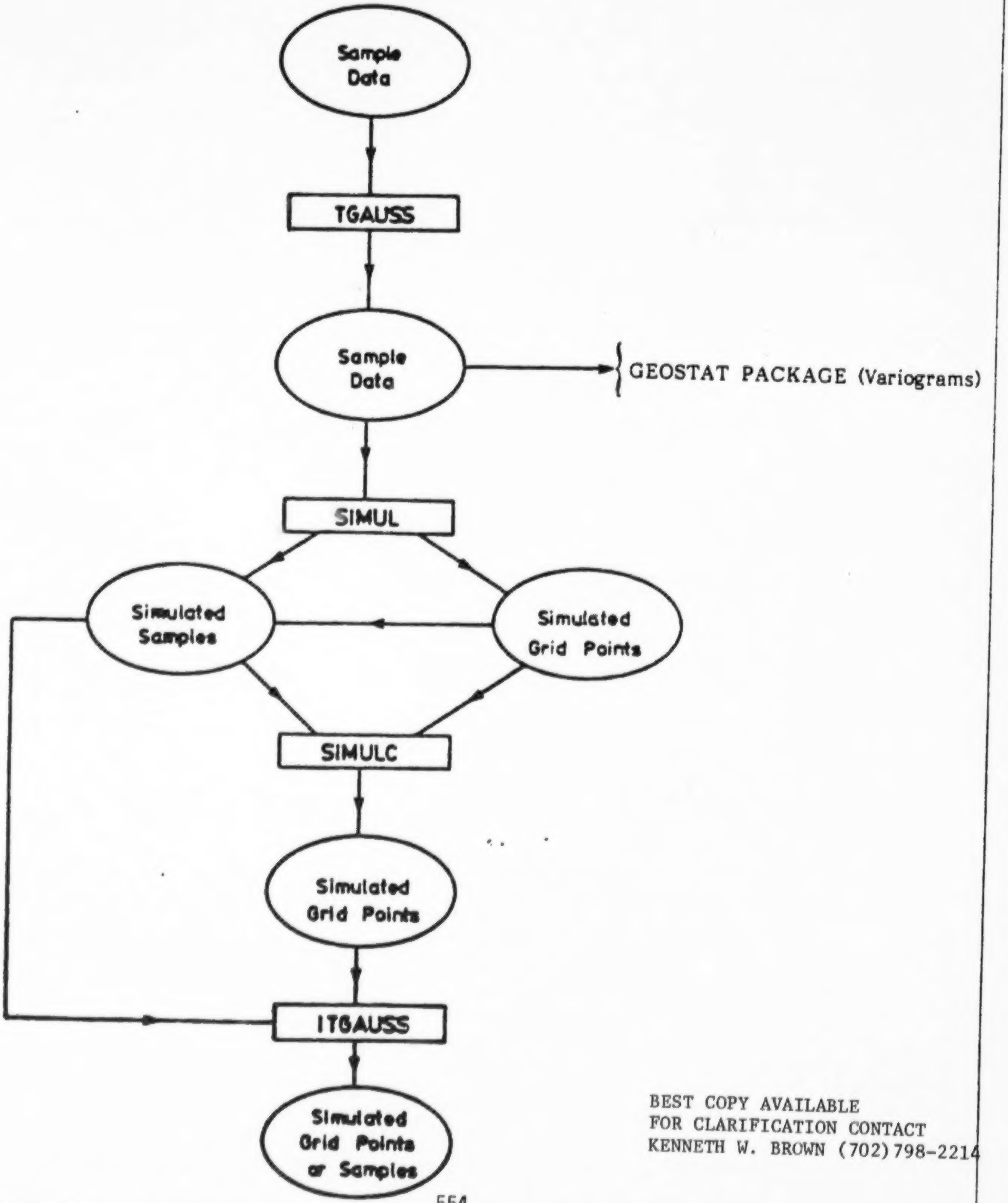
Transformed values are written on an output file. Statistics, and a sorted list of these values can be produced. If they are assigned to grid points, variograms can be computed and a map may be printed. If they are assigned to sample points or points with a specific location, the variograms may be computed in a further step by the MARECX program of the TOXIPAC PACKAGE.

```
**** TRANSFORMATION OF SIMULATED VALUES ****
**** HISTOGRAM OF TRANSFORMED VALUES ****
```



```
**** NORMAL END OF PROGRAM ****
```

FLOWCHART OF THE POLYSIM PACKAGE


Sample Data
TGAUSS
Sample Data
GEOSTAT PACKAGE (Variograms)
SIMUL
Simulated Samples
Simulated Grid Points
SIMULC
Simulated Grid Points
ITGAUSS
Simulated Grid Points or Samples

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APPENDIX N

GEOSTATISTICAL SOIL RESULTS FOR THE DALLAS LEAD STUDY

GEOSTATISTICAL SOIL RESULTS FOR THE DALLAS LEAD STUDY

Kriging Outputs

Environmental contaminants in soil may be inadequately describable in space using classical statistical techniques. Typically, classical statistics assumes that the population of values are distributed as random variables and attempts to describe the mean of these values within some confidence limits. In reality, environmental contaminants in soil may not be randomly distributed but are correlated in space. When spatial correlation exists, samples taken close together agree in magnitude more than samples taken further apart. Kriging uses this correlation structure of soil samples over an area to draw isopleths of pollutant estimates and of the error accompanying these estimates. The spatial information gained form the monitoring activity is more detailed and of known precision.

The enclosed report has six overlays, i.e. a Lead Isovalues Map and a Kriging Errors Map for each study area (REF, DMC, and RSR). The overlays are the same scale (1" = 800') as the aerial photographs used during the sampling phase of the study. There are three street intersections indicated on each overlay so that correct orientation can be made on the appropriate photo.

Overlay of LEAD ISOVALUES

Overlay the three LEAD ISOVALUES maps over the appropriate study area (REF, DMC, and RSR) aerial photo. The labels on the isopleth are in ppm. The DMC and RSR study areas are similar enough in contrast to REF, to be discussed together. Note that on each of them, there are closed nested elliptical curves approximately in the center of each study area. The nestedness shows that there is a rapidly changing gradiant, which implies there is a big change between the inside lead values and the surrounding lead values. This nestedness or change of values tends to delineate the dirty areas. For example, DMC rises from a 300 ppm lead concentration isopleth to an inside isopleth of 3000 ppm and RSR rises from a 300 ppm lead concentration isopleth to an inside isopleth of 2500 ppm. The closed elliptical curve concentration also implies that the primary source is within the curve. For example, if the lead contamination was largely due to the roads (line sources), elongated curves like tubes along the roads would be expected. The maps do not reflect any such open curves. Also, the location of the smelters in each area (DMC and RSR) is in the southern part of the nested closed curves. The location and the shape of the curves along the prevailing wind patterns in Dallas, tend to further support the conclusion that the smelters are the primary sources of lead contamination in the area.

In contrast the REF study area appears to have nested closed curves but they are of lower magnitude and smaller change. For example, the highest nested set of closed curves has a maximum inside concentration isopleth of

500 ppm lead and a concentration change or gradiant of 200 ppm outside to 500 ppm inside.

Both DMC and RSR have a few other nested closed curves which must be evaluated by their magnitude and change. The nested closed curves at the right edge and halfway up the overlay of DMC is due to one very high (10,400 ppm) sample value and thus requires more investigation before a conclusion can be drawn that the total inscribed area represents a true hot spot. The sample bank log book describes this site as heavy industrial.

Overlay of KRIGING ERRORS

The KRIGING ERRORS is like the Error of Estimation in a regression equation or the standard deviation of a sample mean. The isopleth's value from the map must be divided by 10 and take the antilog. In this analysis, KRIGING ERROR is multiplicative instead of the more common additive form because a natural logarithmic transformation was used to normalize the data. That is, the lower confidence limit for the true value of the isopleth is the concentration estimate divided by the kriging error and upper limit is that estimate times the kriging error. For example, the inside maximum isopleth of DMC is 3,000 ppm and overlays KRIGING ERRORS isopleths of 1.5 and 1.8. Taking the larger value, the 1 sigma (68%) confidence interval on the isopleth's true value would not be less than 1,646 (300 1.8) and not more than 5,466 (3,000 x 1.8) (i.e. the true value of lead is between 1,646 and 5,466).

The inside maximum isopleth for RSR is 2,500 ppm and overlays KRIGING ERRORS isopleths of 1.9 and 2.0. Taking the larger value, the 1 sigma (68%) confidence interval, on the isopleth true value would not be less than 1,250 (2,500 2.0) and not more than 5,000 (2,500 x 2 0).

The most important use of the kriging error is to evaluate the adequacy of the sampling activity. Note that all three areas have more or less circular isopleths of kriging error at the edges. These circles are the highest value of the error isopleths, indicating that the sampling activity was comprehensive and left no areas of high error in the areas of interest. Next, note that the boundary Kriging error, or worst case error, is 2.0 for REF and 2.2 for RSR and DMC. Most of the interior error isopleths are small circles of 1.5 or 1.8 values. With a multiplicitive error, a value one is perfect knowledge and is obviously unattainable in real world sampling (Multiplicative Kriging Error > 1.0), and a multiplicative error of 2.0 or 2.2 is not unusual in ecological sampling. Thus, the isomaps of kriging error show that the sampling activity was comprehensive over the area of interest and intense enough to give errors of estimate less than or equal to 2.2.

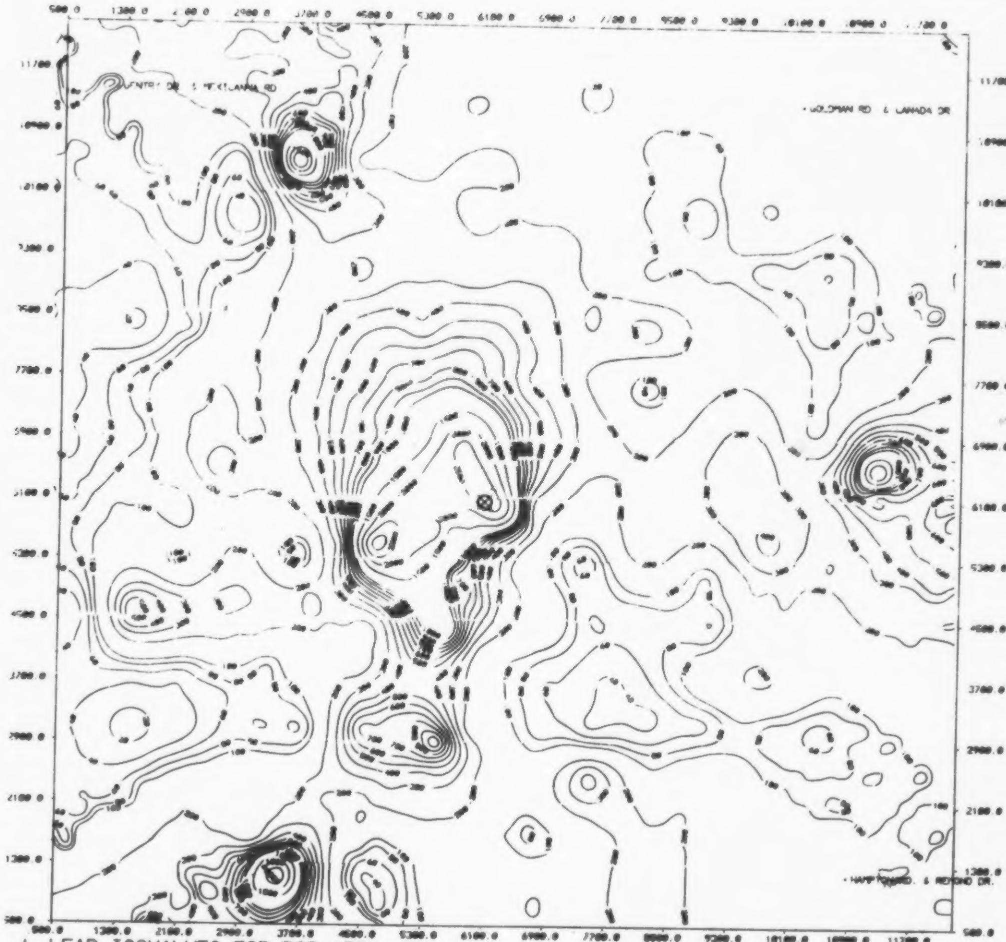
Conclusions

1. The Kriging error isomaps show that the field sampling was comprehensive enough to cover the areas of interest and intense enough to insure an error of estimate of less than 2.2.

2. The patterns of DMC and RSR areas are different from the patterns of the REF area in magnitude of lead values (500 vs. 2,500, 3,000 ppm) and gradients (size of change) between inside and outside lead isopleths (200 to 500 vs. 300 to 2,500, 300 to 3,000 ppm).

3. Among a few patterns of high values both DMC and RSR have a single dominant pattern that includes the smelter. (Dominance is defined as both large area covered and high lead values.) The closure of the pattern implies the source is inside and there is a steep gradiant demarking the polluted area.

4. DMC has a second high pattern at the right edge and halfway up the page that is caused by one data sample (10,400 ppm). This data value probably reflects a local maximum for a very small area and should be verified by reanalysis or resampling before any decision concerning that area is made.


500.0
1300.0
2100.0
2900.0
3700.0
4500.0
5300.0
6100.0
6900.0
7700.0
8500.0
9300.0
10100.0
10900.0
11700.0
GOLDMAN RD. & LAMADA DR
HAMPTON RD. & REMOND DR.
SCALE 1" = 800'

LEAD ISOVALUES FOR RSR AREA

ISOPLETHS OF KRIGING ESTIMATES OF LEAD CONCENTRATIONS (ug/g) IN SOIL AT THE RSR STUDY AREA

⊗ – LOCATION OF THE RSR SMELTER

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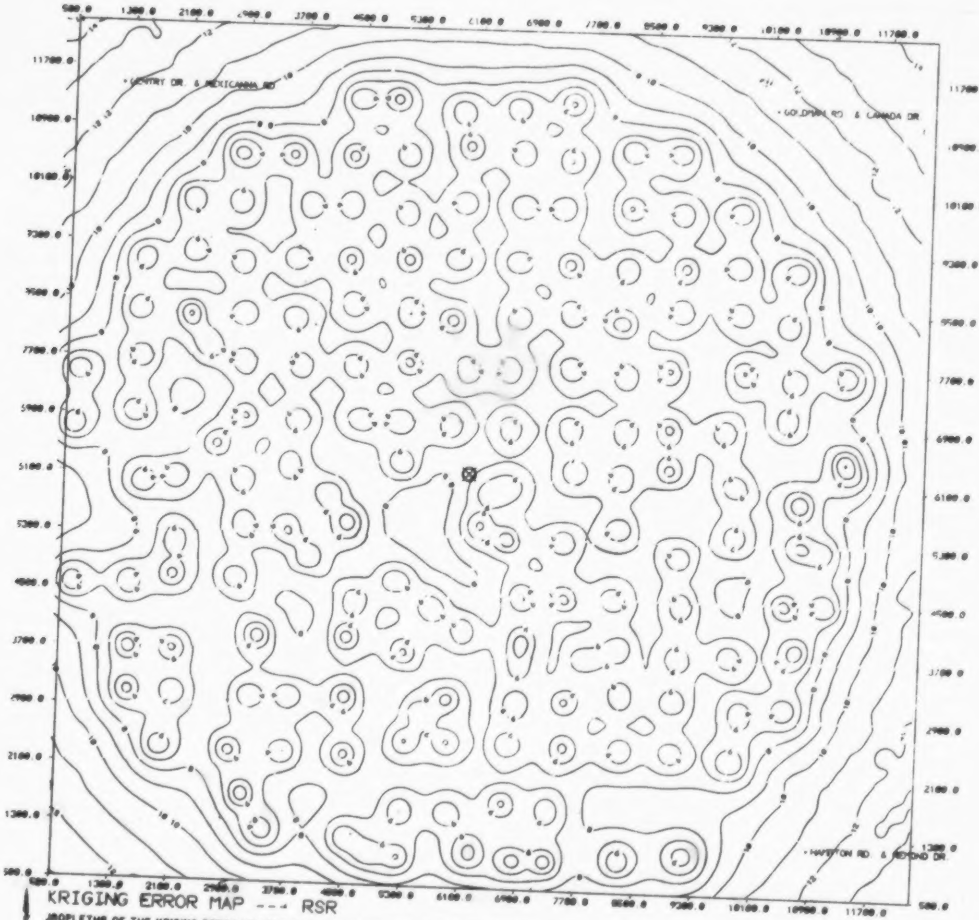
500.0
1300.0
2100.0
2900.0
3700.0
4500.0
5300.0
6100.0
6900.0
7700.0
8500.0
9300.0
10100.0
10900.0
11700.0
CENTRY DR. & MEXICANNA RD
GOLDMAN RD. & CANADA DR.
HAYSTON RD. & BEYOND DR.

KRIGING ERROR MAP --- RSR

ISOPLETHS OF THE KRIGING ERROR OF LEAD CONCENTRATIONS IN SOIL AT THE RSR STUDY AREA
(TEN TIMES THE NATURAL LOG OF THE MULTIPLICATIVE STANDARD DEVIATION)

⊠ - LOCATION OF RSR SMELTER

SCALE 1" = 800'

Multiplicitive Standard Deviation

10 x Log Value (map value)	Antilog Value (circulation value)
4	1.5
6	1.8
7	2.0
8	2.2
10	2.7

e.g. Map value of 4 means:

$\frac{\text{est. lead value}}{1.5} \leq$ true lead value $\leq 1.5 \times$ est. lead value

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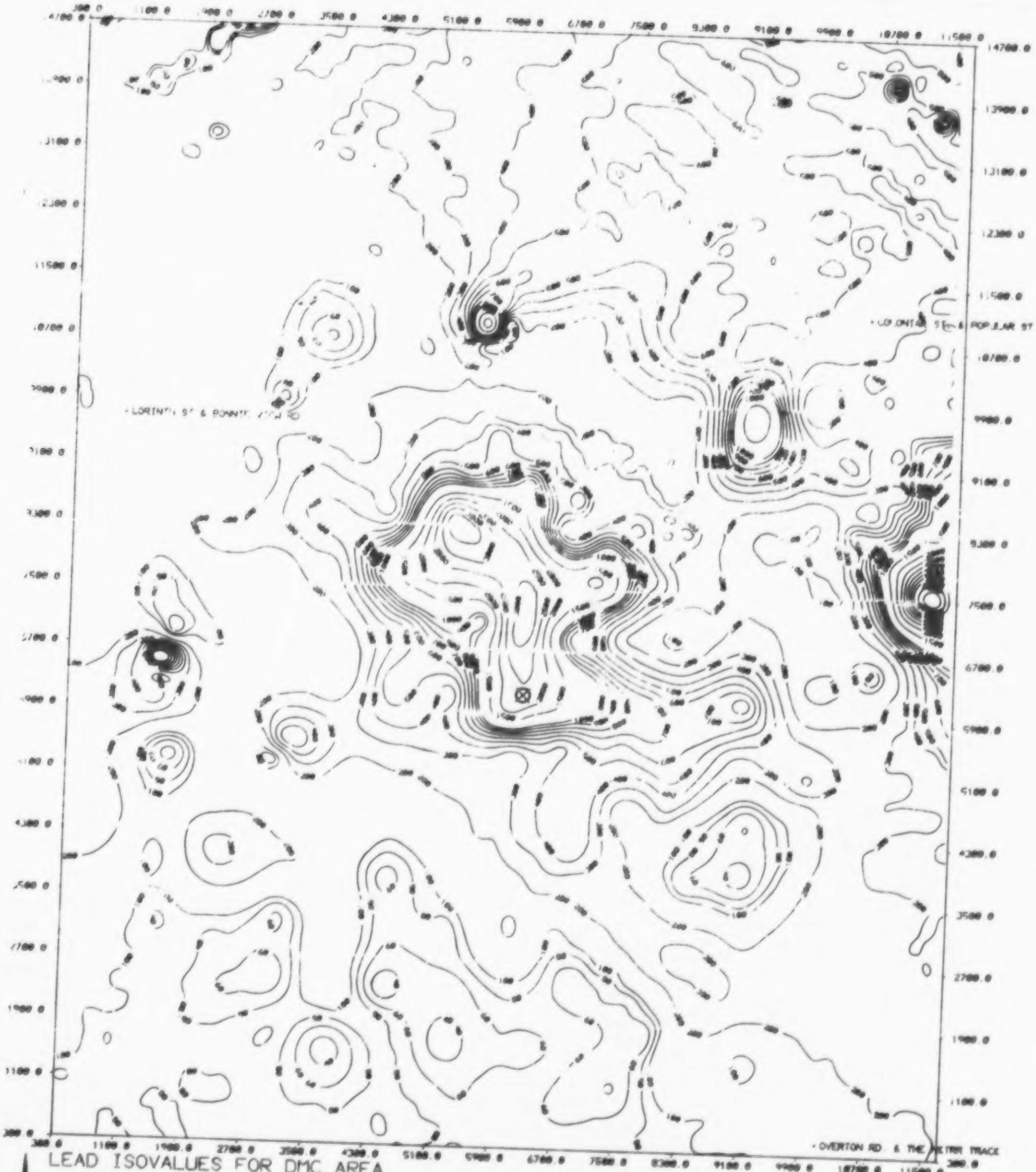
300.0
1100.0
1900.0
2700.0
3500.0
4300.0
5100.0
5900.0
6700.0
7500.0
8300.0
9100.0
9900.0
10700.0
11500.0
14700.0
13900.0
13100.0
12300.0
11500.0
10700.0
9900.0
9100.0
8300.0
7500.0
6700.0
5900.0
5100.0
4300.0
3500.0
2700.0
1900.0
1100.0
300.0
• COLONIAL ST & POPLAR ST
• CORINTH ST & BONNIE VIEW RD
• OVERTON RD. & THE METRR TRACK

LEAD ISOVALUES FOR DMC AREA

ISOPLETH OF KRIGING ESTIMATES OF LEAD CONCENTRATIONS (ug/g) IN SOIL AT THE DMC STUDY AREA

SCALE 1" = 800'

⊗ - LOCATION OF THE DMC SMELTER SCALE 1" = 800'

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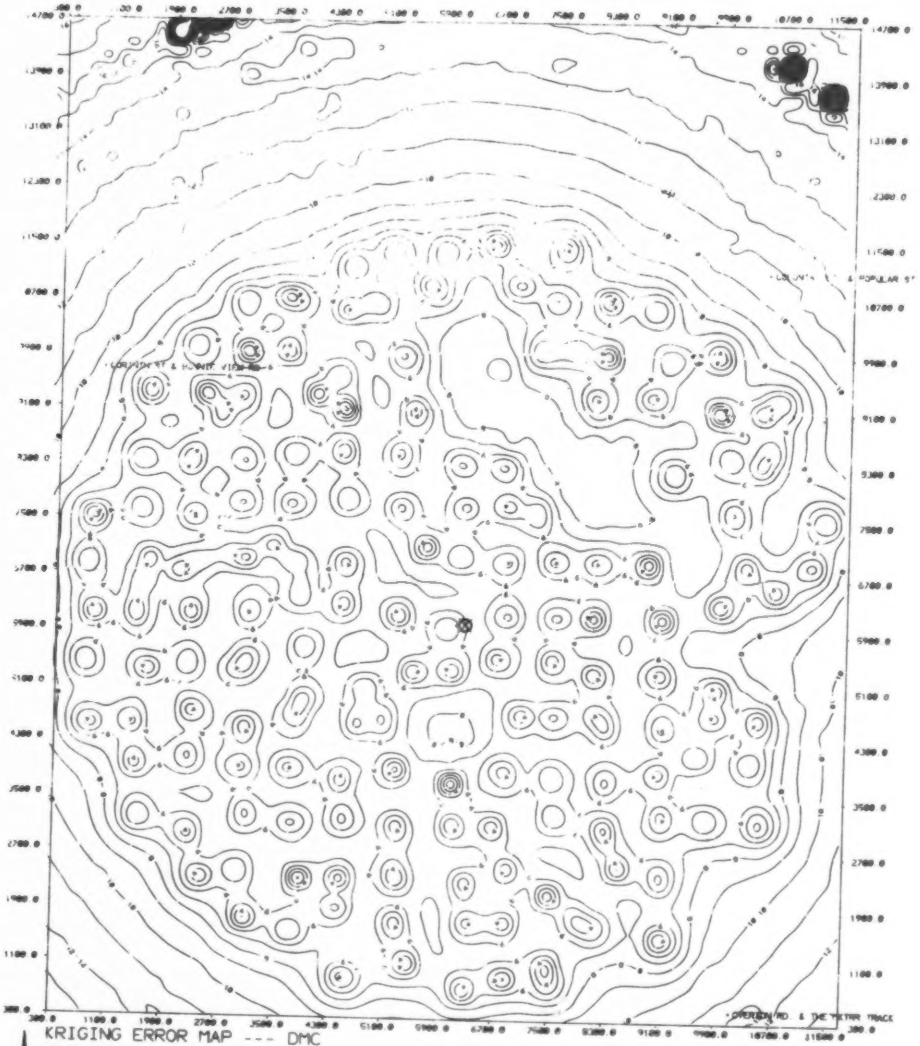
300.0
1100.0
1900.0
2700.0
3500.0
4300.0
5100.0
5900.0
6700.0
7500.0
8300.0
9100.0
9900.0
10700.0
11500.0
14700.0
13900.0
13100.0
12300.0
11500.0
10700.0
9900.0
9100.0
8300.0
7500.0
6700.0
5900.0
5100.0
4300.0
3500.0
2700.0
1900.0
1100.0
300.0

KRIGING ERROR MAP --- DMC

ISOPLETHS OF THE KRIGING ERROR OF LEAD CONCENTRATIONS IN SOIL AT THE DMC STUDY AREA
(TEN TIMES THE NATURAL LOG OF THE MULTIPLICATIVE STANDARD DEVIATION)
⊗ – LOCATION OF THE DMC SMELTER

SCALE 1" = 800'

Multiplicitive Standard Deviation

10 x Log Value (map value)	Antilog Value (circulation value)
4	1.5
6	1.8
7	2.0
8	2.2
10	2.7

e.g. Map value of 4 means:

$$\frac{\text{est. lead value}}{1.5} \leq \text{true lead value} \leq 1.5 \times \text{est. lead value}$$

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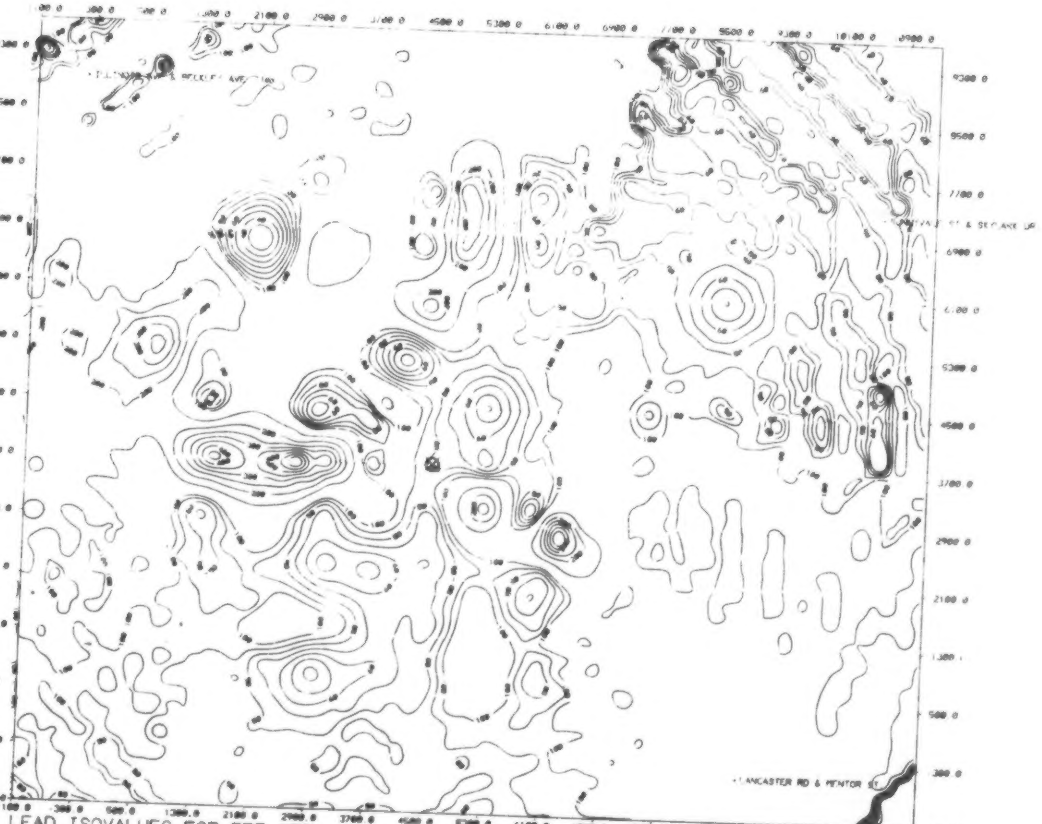

LEAD ISOVALUES FOR REF AREA

ISOPLETHS OF KRIGING ESTIMATES OF LEAD CONCENTRATIONS (ug/g) IN SOIL AT THE REF STUDY AREA

⊗ - LOCATION OF FRIO DRIVE & KEIST BLVD. INTERSECTIONS

SCALE 1" = 800'

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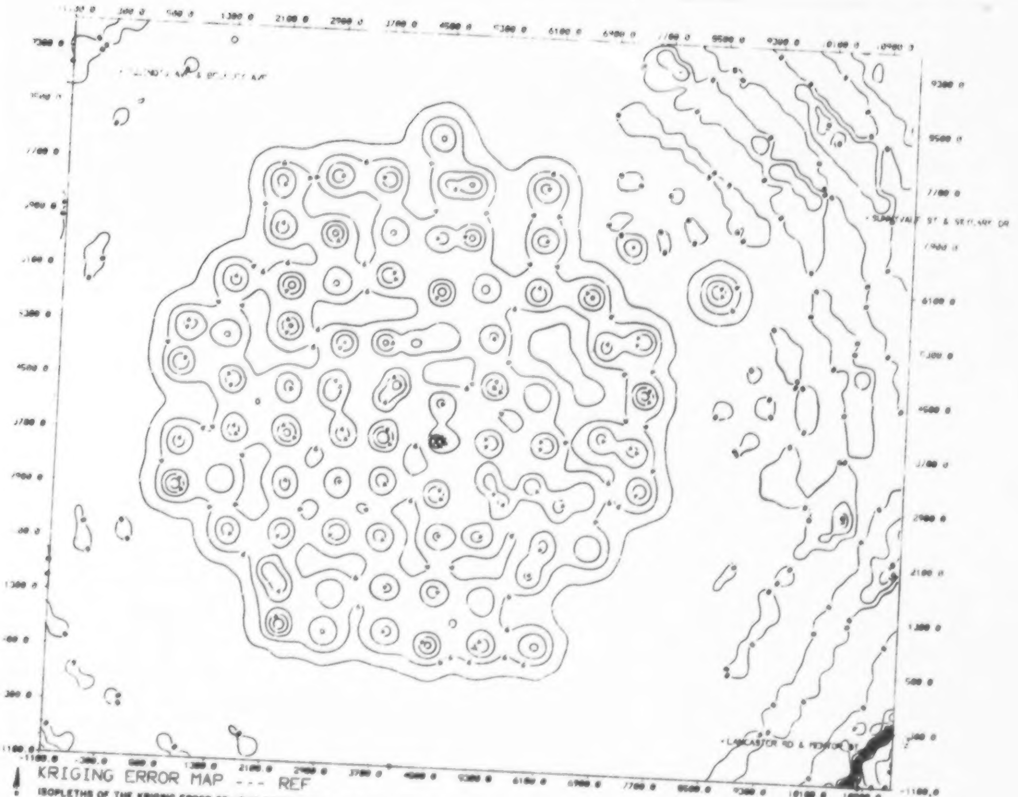

KRIGING ERROR MAP --- REF

ISOPLETHS OF THE KRIGING ERROR OF LEAD CONCENTRATIONS IN SOIL AT THE REF STUDY AREA
(TEN TIMES THE NATURAL LOG OF THE MULTIPLICATIVE STANDARD DEVIATION)
⊗ - LOCATION OF FRIO DRIVE & KEIST BLVD. INTERSECTIONS

SCALE 1" = 800'

Multiplicitive Standard Deviation

10 x Log Value (map value)	Antilog Value (circulation value)
4	1.5
6	1.8
7	2.0
8	2.2
10	2.7

e.g. Map value of 4 means:

$\frac{\text{est. lead value}}{1.5} \leq$ true lead value $\leq 1.5 \times$ est. lead value

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